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LABORATORY MANUAL  
OF  
DYEING  
AND  
TEXTILE CHEMISTRY

BY  
J. MERRITT MATTHEWS, PH.D.

*FIRST EDITION*  
FIRST THOUSAND



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## PREFACE.

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THIS volume is designed with the purpose of meeting the demand for a text-book on the subject of textile dyeing and chemistry. The method of presentation and the subject matter herein contained have been the outcome of a number of years of teaching on the part of the author. Care has been taken to avoid a too purely scientific generalization, else as a text-book it would lose its main value. The subject treated is a technical one, and an endeavor has been made to present it in a technical manner; that is to say, definite facts have been presented in a definite form. For this purpose, the experimental method has been adopted, further elucidation being given in additional notes as succinctly expressed as possible. The insertion of quiz questions has also been carried out in order to stir up the *thought* of the student after he has performed the *manual* experiment.

This book is intended as an elementary manual for the students who are primarily to be found in the various textile schools and in institutions where the many branches of technical chemistry are taught. It is hoped that it will be of some value to both the student and the teacher interested in this line of instruction. It is primarily a *laboratory* manual or guide, and is intended to direct and supplement the lectures of the teacher, and should be used in conjunction with the more general treatises on the subject.

The subject matter is divided in such a manner as to provide a course of instruction for one complete college year, each section corresponding to a weekly apportionment of study. The experimental part will require from eight to twelve hours of laboratory practice each week; though sufficient experiments have been given to allow the teacher considerable latitude as far as selection of material and length of laboratory practice are concerned.

J. MERRITT MATTHEWS.

TAUNTON, MASS., *December*, 1908.



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# Laboratory Manual of Dyeing and Textile Chemistry

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## INTRODUCTORY.

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1. **Apparatus Required.** — In carrying out the dye-tests herein described, it will be found convenient to employ skeins of wool and silk weighing 5 grams, and of cotton 10 grams. The dye-baths should contain about 300 to 400 cc. of water, and should be of porcelain, glass, or enameled iron-ware. A good form of experimental dye-bath is that shown in the illustration (Fig. 1). It consists of a round copper vessel lined inside with asbestos, and provided with a perforated iron bottom. Its top contains four openings through which the dye-pots are inserted. This copper air-bath is placed on an iron stand provided with a gas burner. The dye-pots are of porcelain and are held by beveled copper collars with wooden handles. The air-bath is so arranged that when the dye-pots are in position they are raised about an inch above the bottom plate. Such a dye-bath allows of a uniform heating of the four pots, and the temperature may be raised rapidly or slowly at will, by regulation of the gas flames, and it is an easy matter to bring the liquid in the pots to an active boil. There are other forms of experimental dye-baths in use where solutions of calcium chloride, common-salt, glycerin, etc., are used for heating the dye-pots. Strong solutions of calcium chloride are capable of being heated far above the boiling-point of water, and consequently in such a bath it is easy to bring the dye-pots to the boil. But calcium chloride solutions attack the baths in which they are contained. In case of copper vessels with soldered

seams the solder is rapidly eaten out and leaks frequently occur. In a dye-bath using a solution of calcium chloride the seams should be brazed, which makes the apparatus rather expensive, and even then the copper itself is soon attacked. With solutions

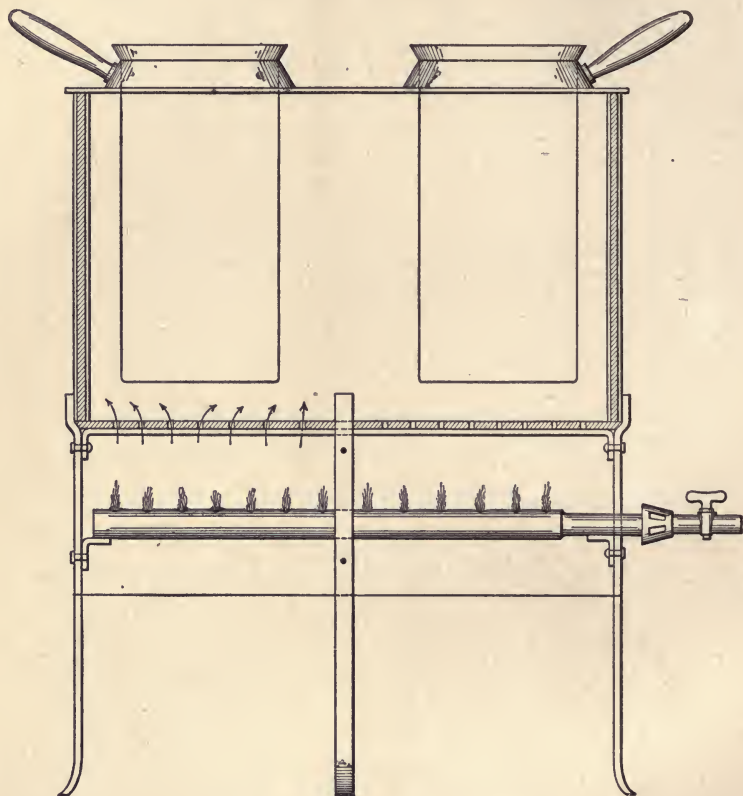


FIG. 1. — Experimental Dye Bath.

of common-salt it is difficult to obtain a temperature of  $212^{\circ}$  F. in the dye-pots; that is, to bring them to a state of active boiling. A temperature of  $210^{\circ}$  F., however, can be maintained, and probably this is nearer the actual temperature of the open dye-vat in practice, and gives as good results as if the liquid was in an actual state of ebullition. Solutions of common-salt are



perhaps to be preferred to those of calcium chloride, as they do not have as corroding an action on the copper dye-bath. By the use of glycerin in the bath a boiling temperature can readily be obtained in the dye-pots, but glycerin baths continually emit disagreeable vapors. Whenever possible, baths containing such solutions should be heated by a steam-coil (with steam under pressure) rather than by direct gas flames. The great disadvantage of all baths using solutions, and one from which the air-bath is free, is that the water is constantly being evaporated from the solution and has to be as constantly replaced.

When dyeing the test-skeins they should be systematically "worked" or turned in the dye-solution. This is best accomplished by suspending the skein in the bath from two glass rods, and using these from time to time for the purpose of turning the skeins. These glass rods should be one-fourth to three-eighths inch in diameter, and 8 to 10 inches in length. The skeins should be turned sufficiently to insure even penetration of the solution through the entire portion of the material.

The dyestuffs and various chemicals employed in carrying out the dye-tests should be used in the form of solutions of such strength that small quantities of the products may be measured out in convenient volumes. As the amount of material being dyed (5 or 10 grams) is relatively small, and as small amounts of the dyestuffs, etc., are used, it would be both inconvenient and inaccurate (unless very precise weighings were made on expensive and accurate balances) to weigh the chemicals employed in each test; but by preparing solutions of definite strengths the required amounts may be readily and accurately measured off. The proper preparation of these solutions will be taken up as demanded by the course of the experiments. For the measurement of the solutions a glass cylinder graduated into 100 cc. is very convenient; this readily permits of the rather accurate measurement of such quantities as 5 cc., 10 cc., etc. In cases where very minute quantities are desired, and it is necessary to measure to an accuracy of  $\frac{1}{10}$  cc., a small glass tube (known as Mohr's pipette) accurately graduated to  $\frac{1}{10}$  cc. is very useful.

These pipettes may be obtained in sizes holding 5, 10, 25, or 50 cc., and by their use volumes accurate to  $\frac{1}{10}$  cc. may be readily measured out. A thermometer is also necessary for use in the dye-tests. A good, practical, and inexpensive form is the so-called "dairy" thermometer with a paper scale and reading to 220° F. By the use of this thermometer the temperatures of the dye-solutions or other liquids employed in the tests may be ascertained. An agate cup (pint or quart size) is a useful adjunct for the preparation and mixing of the various solutions needed. A bunch of small tags should also be available so that every skein with which a test has been made may be properly labelled for identification and reference.\*

\* The following is a list of apparatus supplied as an equipment to each individual student in the dye laboratory of the Philadelphia Textile School:

- One dye-bath (for four pots).
- Four porcelain beakers (320 cc.).
- Four copper collars (with wooden handles).
- Twelve glass rods ( $\frac{1}{4}$  inch diameter by 8 inches in length).
- One dairy thermometer (to 220° F).
- One graduated cylinder (100 cc.).
- One Mohr's pipette (10 cc. divided into tenths).
- One agate cup (pint).
- One bunch tags (with white strings).

## SECTION I.

### CHEMICAL STUDY OF THE FIBRES.

**Experiment 1. Action of Acids on Wool and Cotton.** — Place about 300 cc. of water in one of the porcelain beakers employed for the dye-tests, and add 2 cc. of concentrated sulphuric acid. In this "bath" boil a test-skein of woolen yarn together with one of cotton yarn for 20 minutes; then remove the skeins, squeeze out the excess of liquid, and dry without washing. After drying test the strength of the two skeins, and it will be found that the cotton (1)\* has been very much weakened and may be easily pulled apart, whereas the wool (2) does not appear to have been much affected. Boil a second set of woolen and cotton skeins in the same acid bath for 20 minutes, then wash well in several changes of fresh water. Take the woolen skein and, together with another one of untreated wool, dye by boiling for 20 minutes in a beaker containing 300 cc. of water and 10 cc. of Acid Magenta solution (containing 5 grams of the dissolved dyestuff per liter); then wash well and dry. It will be noticed that the skein which has been treated with acid (3) will be dyed a heavier color than the second skein (4). This is due to the wool having combined chemically with the acid in its first treatment, thus allowing it to react more readily with the acid dyestuff employed. Take the second cotton skein and pass it through a cold solution of 1 gram of soda ash in 300 cc. of water for 10 minutes; then rinse in fresh water and dry. It will be found that this skein (5) has not become weakened by the treatment

\* The parenthetical numbers refer to samples to be taken from the tests by the student, and correspond to the sample numbers given in a list at the end of each section. These samples should be neatly and systematically mounted in a book specially prepared to receive them, and each one should be properly numbered and labelled, and should have the careful criticism of the instructor.



with the acid solution, as the latter has been neutralized by the alkali before drying.

**Experiment 2. Action of Organic Acids on Cotton.** — Work a test-skein of cotton yarn in a bath containing 300 cc. of water and 5 cc. of acetic acid for 20 minutes at a temperature of 160° F. Squeeze and dry without washing. Test the strength of the dried skein (6) and it will be found not to have become much weakened. Acetic acid is a volatile organic acid and on drying is volatilized from the fibre.

**Experiment 3. Action of Alkalies.** — Boil a skein of woollen yarn together with one of cotton in a bath containing 300 cc. of water and 10 cc. of caustic soda solution (60° Tw.). The wool will be disintegrated and dissolved (7). Wash and dry the cotton skein and it will be found not to be appreciably altered (8). Repeat the test, using 10 cc. of a solution of sodium carbonate instead of caustic soda. Boil for 20 minutes, then wash and dry. It will be found that the wool has become much weakened and is lifeless and dull in appearance (9), while the cotton is not changed (10). Repeat this test, using 10 cc. of a solution of ammonium carbonate; boil for 20 minutes, then wash and dry. It will be noticed that in this case neither the wool (11) nor the cotton (12) is affected in strength.

**Experiment 4. Action of Metallic Salts (Mordants).** — Boil a skein of wool together with one of cotton for 20 minutes in a bath containing 300 cc. of water, and 10 cc. of chrome solution. Rinse with fresh water. Then boil the woollen skein together with another of untreated wool in a bath containing 300 cc. of water and 20 cc. of a solution of madder. Finally wash well and dry. It will be found that the untreated skein (13) has not become dyed, whereas that treated with the chrome has become colored (14). Take the cotton skein which has been treated with the chrome and boil it also in a solution containing 300 cc. of water and 20 cc. of madder solution, then wash well and dry (15). It will be found that the cotton skein has taken up but very little dyestuff, as this fibre absorbs but a small amount of the mordant. Madder is a dye which has no direct affinity for the fibres, but it

forms a color-lake with metallic salts such as chrome; hence the unmordanted wool did not become dyed. Due to the fact that wool has a much greater affinity for metallic salts than cotton, it will be noticed that the former fibre is dyed much deeper than the latter.

**Experiment 5. Action of Bleaching Powder.** — Steep a skein of woollen yarn together with one of cotton in a cold solution of bleaching powder of about 2° Tw. strength for 30 minutes. Then pass into a cold bath containing 300 cc. of water and 10 cc. of a dilute solution of hydrochloric acid (the odor of what gas is noticed here) and work for 10 minutes. Then wash well in fresh water. It will be found that the cotton (16) has become bleached, but that the wool (17) has acquired a deeper yellow tint and is harsh in feel after drying. The wool has combined with the chlorine of the bleaching powder in a chemical manner while the cotton has not; the only effect in the latter case being that the bleaching liquor destroys the coloring-matter naturally present in the cotton. Next, take this skein of “chlorinated” wool together with a skein of untreated wool and dye them for 20 minutes at 160° F. in a bath containing 300 cc. of water and 5 cc. of a solution of Diamine Sky Blue; wash and dry. It will be found that the “chlorinated” skein (18) takes up much more dyestuff than the other skein (19) and is dyed a darker shade. Next, take portions of these two skeins and plait them together and steep in a small quantity of warm soap solution and rub vigorously between the hands to imitate the action of fulling or milling. It will be found that the ordinary wool (20) will readily felt together, while the chlorinated wool (21) does not.

#### NOTES.

**1. Action of Acids on Wool and Cotton.** — The animal and vegetable fibres show a marked contrast in their behavior with acids. Wool absorbs mineral acids (sulphuric, hydrochloric, and nitric) from solution and, unless the acid is quite concentrated, the fibre is not decomposed. The acid, in this case, no doubt combines chemically with the wool on account of the *basic* nature of this fibre. This is evidenced by the fact that wool which has

been treated with acid will dye with acid coloring-matters much better than ordinary wool. Also, when wool is treated with a solution containing sulphuric acid and then washed until the wash waters are neutral, there will still be some of the acid remaining in the wool. Cotton, on the other hand, is rather easily affected by solutions of the mineral acids, especially when such a solution is allowed to dry into the fibre. Cotton does not possess any basic qualities, and therefore does not combine chemically with the acid, thereby neutralizing it, as in the case of wool. Unless employed in very weak solutions, all the mineral acids have a tendering action on cotton, causing a disintegration of the fibre through a breaking down of the cellulose molecule of which the cotton is composed. The compound of cellulose so formed is known as *hydrated cellulose* and is brittle in nature. On this difference in the reaction of wool and cotton with acids is based the process of "carbonizing" or separating vegetable fibres from wool in woven fabrics or in shoddy where it is desired to recover the wool and eliminate the cotton. Organic acids (such as formic, acetic, oxalic, and tartaric) do not have the same tendering action on cotton as the mineral acids; formic and acetic acids, being both volatile, are removed from the fibre on drying and hence do not injure cotton; oxalic and tartaric acids, on the other hand, are not volatile, and if strong solutions are used somewhat tender the cotton when drying.

**2. Action of Alkalies on Wool and Cotton.** — Alkalies react with the animal and vegetable fibres in just the opposite manner to acids. Caustic soda, even in very dilute solutions and at not very high temperatures, will completely disintegrate and dissolve the wool fibre; whereas cotton is not affected. Even with solutions of sodium carbonate (soda ash) the wool fibre will be seriously weakened and injured in appearance unless such solutions are comparatively weak and employed at rather low temperatures. Due to these facts caustic soda cannot be used for the scouring of wool, nor should it be used in any connection with wool. Soda ash when employed in scouring or any other process in contact with wool, must be carefully handled in order that the



solution of the same does not become too concentrated nor heated too high. Cotton, on the other hand, is scoured by the use of boiling caustic soda or soda ash without fear of being injured. Ammonium carbonate and ammonia water are much milder in their alkaline action and do not have any injurious effect on wool at ordinary concentrations, on which account they make very good scouring compounds, although too expensive for the majority of materials.

**3. Action of Metallic Salts on Wool and Cotton.** — Wool is quite reactive towards solutions of the majority of metallic salts, absorbing the most of them from solution and fixing the oxide of the metal in chemical combination with the fibre. For instance, when wool is boiled with a dilute solution of potassium bichromate (chrome), the latter salt becomes decomposed to a considerable extent and quite a proportion of chromium oxide becomes chemically combined with the fibre. This fact is evidenced by the wool showing the presence of the metallic compound by its color and by being able to form a color-lake with certain dye-stuffs which will not combine directly with ordinary wool. Cotton, on the other hand, has but very slight affinity for metallic salts, being very inert in this connection, as the fibre does not appear to be able to absorb and fix the metallic oxide as in the case of wool. This action of wool with metallic salts forms the basis of the operations of *mordanting* wool preliminary to the dyeing with the alizarin and other mordant colors which require a base of some metallic oxide with which to form a color-lake; and also due to the inert character of cotton, this fibre cannot be readily dyed with these colors. Bleaching powder has a peculiar action on wool; this chemical is a strong oxidizing agent and in hot solutions of any considerable concentration will rapidly disintegrate the wool fibre. In cold and dilute solutions, however, a chemical combination apparently takes place between the wool and the chlorine evolved by the bleaching powder, giving a product known as "chlored" or "chlorinated" wool without much physical alteration of the fibre. Wool so treated exhibits a much stronger affinity towards many coloring-matters,

and almost completely loses its felting properties, and acquires a higher luster. Cotton does not combine with the chlorine of the bleaching powder, but shows the strong oxidizing action of the latter in becoming bleached. This reaction is the basis of the method of bleaching cotton materials.

### SAMPLES.

1. Cotton treated with mineral acid; showing weakness.
2. Wool treated with acid; not weakened.
3. Wool treated with acid and dyed.
4. Wool not treated with acid and dyed.
5. Cotton treated with mineral acid, then neutralized with an alkali; not weakened.
6. Cotton treated with organic acid; not weakened.
7. Wool boiled with caustic soda; dissolved.
8. Cotton boiled with caustic soda; not affected.
9. Wool boiled with solution of soda ash; much injured.
10. Cotton boiled with solution of soda ash; not affected.
11. Wool boiled with solution of ammonium carbonate; not injured.
12. Cotton boiled with solution of ammonium carbonate; not affected.
13. Unmordanted wool dyed with madder.
14. Wool treated with chrome and dyed with madder.
15. Cotton treated with chrome and dyed with madder.
16. Cotton treated with bleaching powder; becomes bleached.
17. Wool treated with bleaching powder; becomes yellowish.
18. Chlorinated wool dyed.
19. Unchlorinated wool dyed for comparison.
20. Unchlorinated wool fulled with soap.
21. Chlorinated wool fulled with soap.

### QUIZ I.

1. What is the effect of allowing acid solutions to dry in the cotton fibre? How does wool differ in this respect?
2. After treatment with solutions containing non-volatile acids, why should cotton be very thoroughly washed before drying?
3. What is the difference in the chemical nature of organic and mineral acids? Name several representatives in each class.
4. Why do solutions of acetic acid not have the same weakening action on cotton as solutions of sulphuric acid? Does oxalic acid behave any differently from acetic acid and why?
5. Why is it preferable to use acetic acid in connection with cotton rather than sulphuric acid?



6. Does wool combine in a chemical manner with sulphuric acid? How can this be shown?

7. If it is necessary to treat cotton with acid solutions, how may the injurious effects of the acid be prevented?

8. What is the action of dilute solutions of caustic soda on wool? On cotton?

9. Can wool be boiled with soda ash solutions without injury?

10. Do boiling solutions of soda ash have any injurious action on cotton?

11. How do solutions of ammonium carbonate react with wool and cotton?

12. Why should soaps used in scouring wool be free from caustic alkali?

13. Solutions of soda ash are largely used for scouring wool; why should not such scouring baths be used at a boiling temperature?

14. What is meant by a mordant?

15. What is the action of solutions of potassium bichromate on wool? Explain the chemical reaction which takes place.

16. Metallic salts of what character are useful as mordants for wool? What is meant by the "dissociation" of a salt?

17. How does mordanted wool react with a solution of madder?

18. How does cotton differ from wool in its action towards solutions of metallic salts?

19. Why are coloring-matters employed with a metallic mordant more useful on wool than on cotton?

20. What is bleaching powder? Which of its components is the active bleaching agent?

21. What is meant by a degree Twaddle? Explain how densities are determined with a Twaddle hydrometer.

22. The odor of what gas is noticed when the solution of bleaching powder is brought in contact with hydrochloric acid? Give the chemical reaction taking place.

23. Describe the effect of bleaching powder solutions on wool. Why cannot this substance be employed for bleaching wool?

24. What is the action of bleaching powder on cotton?

25. What is meant by "chlorinated" wool? How does such wool differ from ordinary wool in its behavior with dyestuffs?

26. What effect has the chlorination of wool on its felting properties?

## SECTION II.

### SCOURING THE TEXTILE FIBRES.

#### **Experiment 6. Scouring Raw Wool by the Emulsion Process. —**

Weigh out 10 grams of raw wool (22) and scour it in a bath containing 300 cc. of water, 5 grams of soda ash, and 2 grams of soap. Have the soap thoroughly dissolved before adding it to the bath. Work the wool gently at 140° F. for one-half hour, or until it seems thoroughly cleansed. Wash well in fresh warm water to remove all soapy liquor. Dry (23) and reweigh. Calculate the percentage of loss or "shrinkage." In working the wool in the scouring bath care should be taken not to agitate the fibres too vigorously or the wool will become matted or felted together.

**Experiment 7. Use of Potash in Scouring Wool. —** Prepare a scouring bath containing 300 cc. of water, 5 grams of pearl ash (potassium carbonate or potash), and 2 grams of soap. Scour a 10-gram sample of the same wool as used above and proceed in the same manner. Wash well in warm water, allow to dry (24) and reweigh. Compare the two samples thus scoured by the use of the two alkalis.

**Experiment 8. Effect of High Temperatures in Scouring. —** Use the same bath as employed in Exp. 6 and scour another 10-gram sample of the same kind of raw wool, but bring the bath to the boil for one-half hour. Rinse as before in warm water, and allow to dry (25). Reweigh and calculate the percentage of loss, and also compare the general appearance and "feel" of the wool with that scoured in the first experiment.

**Experiment 9. Effect of Using Excessive Alkali in Scouring Raw Wool. —** Scour a 10-gram sample of raw wool in a bath containing 300 cc. of water and 20 grams of soda ash. Work for one-

half hour at a temperature of  $140^{\circ}$  F.; then wash well in warm water and allow to dry (26). Calculate the percentage of loss, and compare the general appearance and feel with the samples scoured by the use of less alkali.

**Experiment 10. Scouring Woolen Yarn by the Usual Method.** — Prepare a bath containing 300 cc. of water, 10 grams of soap, and 2 grams of soda ash. Scour a weighed test-skein of woolen yarn (27) in this bath for one-half hour at a temperature of  $140^{\circ}$  F., then wash in fresh water and allow to dry (28). Reweigh after drying and calculate the percentage of loss due to scouring.

**Experiment 11. Scouring Woolen Yarn Containing Iron.** — Yarn of this nature is best scoured in baths containing only soap, as soda ash or potash will form an insoluble compound with the iron in the fibre which cannot be removed, and which will cause the yarn to dye up dull. Scour a test-skein of woolen yarn containing iron (29) in the same bath as employed for the previous experiment and in the same manner; wash well and dry (30). Scour a second skein of similar yarn in a bath containing 300 cc. of water and 10 grams of soap for one-half hour at  $140^{\circ}$  F.; wash well and dry (31). Compare the appearance of the two scoured skeins.

**Experiment 12. Scouring Cotton with Caustic Soda.** — Prepare a bath containing 5 grams of caustic soda to 300 cc. of water, and boil a skein of cotton yarn (32) therein for one-half hour; then wash in fresh water until all trace of the caustic soda has been removed from the cotton and dry (33). Weigh the skein before and after the scouring and calculate the percentage of loss.

**Experiment 13. Scouring Cotton with Soda Ash.** — Prepare a bath containing 5 grams of soda ash and 300 cc. of water, and boil a skein of cotton yarn therein for 1 hour. Wash well in fresh water and dry (34). Weigh the skein before and after scouring and calculate the percentage of loss. Compare this skein with that in the preceding test as to amount of loss, color, softness, etc.



**Experiment 14. Scouring Cotton with Soap.** — Prepare a bath containing 5 grams of soap and 300 cc. of water, and boil a weighed skein of cotton yarn therein for one-half hour. Wash in fresh water and dry (35). Reweigh and calculate the percentage of loss. Compare this skein with the others of the above experiments.

**Experiment 15. Scouring Cotton with Fankhausine.** — Prepare a bath containing 2 cc. of Fankhausine (50 per cent solution) and 300 c.c. of water. Work a skein of cotton yarn in this bath for one-half hour at 180° F.; then wash and dry (36). Weigh the skein before and after scouring and calculate the percentage of loss. Compare the skein with others in the previous experiments as to color, softness of feel, etc.

**Experiment 16. Scouring of Raw Silk.** — The fibre proper of raw silk is covered with a glue-like material known as sericin. The presence of this latter substance makes raw silk harsh and stiff and without lustre. Boiling soap solutions remove the sericin without affecting the fibre proper of the silk. The scouring of silk is known as "boiling-off," "stripping," or "degumming." The silk will lose about 25 per cent. in weight if completely boiled off. Take a weighed skein of raw silk yarn (37) and boil it for one hour in a solution containing 250 cc. of water and 25 grams of olive oil hard soap; then wash well in fresh warm water and dry (38). Reweigh and calculate the percentage of loss. As a rule, to completely degum the silk it is necessary to boil in several soap baths. Notice the difference in the appearance and "handle" of the boiled-off silk. Stretch and squeeze the dried boiled-off skein so as to soften up the fibre and lustre it (39). It will be found that most of the coloring-matter of the raw silk is in the sericin and is removed in the boiling-off. The soap liquor left after boiling-off the silk is known as "boiled-off liquor," and is used extensively as an addition to the dye-bath in the general dyeing of silk. The soap employed for scouring silk should be of the best quality and as neutral as possible.

## NOTES.

**1. Impurities in Raw Wool.** — The raw wool fibre as it exists in the fleece contains a large amount of natural impurities. These are as follows:

- a. Grease, or wool fat.
- b. Suint, or dried-up perspiration.
- c. Dirt, consisting of dust, sand, burrs, etc.

It is the object of scouring to remove these impurities and leave the fibre pure and clean without material injury to its good qualities. The *greasy matters* in the fleece, known as *wool-fat*, are insoluble in water but are readily emulsified by solutions of soaps or alkalis. Wool-fat differs from most other animal fats in chemical constitution in that the latter are compounds of various fatty acids with glycerin (and hence are called *glycerides*). These rather easily react with caustic alkalis to form soluble *soaps*, a reaction which is termed *saponification*. Wool-fat, however, is not a glyceride, but contains a substance known as *cholesterol* (a body belonging to the general class of alcohols), and does not form soaps with the caustic alkalis. It does, however, form emulsions with more ease than do the other animal fats, and on this account it is rather easily removed from the fibre. The *suint* (a French word for sweat) consists of various metallic salts of organic acids, such, for instance, as potassium acetate. These salts are soluble in water and hence are easily removed in the scouring. The miscellaneous *dirt* in the wool is not soluble in water and is simply mechanically removed by the agitation of the wool in the process of scouring. The amount of impurities in raw wool varies quite largely with the character of the sheep and the locality in which they are grown. Generally speaking, the total impurities may be said to vary from 50 to 80 per cent. of the weight of the fleece, with a general average of about 65 per cent. As a rule, the finer the staple of the wool, the greater amount of grease it will contain; whereas in coarse wools the amount of grease is usually relatively

much less. The loss in weight that wool undergoes on scouring is termed its *shrinkage*, and forms an important item in judging the value of raw wools.

The chemicals chiefly employed in the usual method of scouring wool are soda ash (sodium carbonate) and soaps. Potash (potassium carbonate) is sometimes used, but as it is much more expensive than soda ash its use is more restricted. Most frequently a mixture of soda ash and soap is used, the relative amounts depending on the quality of the wool to be scoured and the amount and nature of the impurities present. As the wool fibre is rapidly injured by solutions of alkalis at high temperatures, the scouring of wool should be carried out at as low a temperature as permissible with perfect removal of the impurities. The temperature of the scouring bath under ordinary conditions should not exceed  $140^{\circ}\text{F.}$ , and in the case of fine lustre-wools the lower temperature of  $100^{\circ}$  to  $120^{\circ}\text{F.}$  is used. Attention has already been drawn to the fact that wool is easily injured by even quite dilute solutions of caustic alkalis, consequently the presence of such is especially deleterious. On this account, the soaps and other ingredients used in the scouring solutions should be free from any very appreciable amount of caustic alkali. After removal from the soap solution, the scoured wool should be thoroughly cleansed from alkali and soap by washing in water. If this is not done the soap will dry into the fibre, and subsequently be very difficult to properly remove. The presence of soapy matters in the wool leads to many bad effects; it causes the product to have a sticky and greasy feel, produces unevenness in dyeing, and when brought into an acid solution (as is generally the case in the application of most dye-stuffs to wool), the soap is decomposed with the liberation of free fatty matter in the fibre, which is a very objectionable result.

**2. The Emulsion Process of Scouring.** — The ordinary process of scouring wool by the use of solutions of soaps and alkalis is called an "emulsion" process on account of the fact that the soapy and alkaline liquors form an emulsion with the greasy matters in the wool. A distinction must be made between an



*emulsion* and a *solution*. An emulsion is an intimate mixture of greasy or oily matters with water (or solution of soap or alkali) in which the grease exists disseminated throughout the mixture in a very finely divided state; usually in the form of minute globules. The emulsion is considered as permanent when the greasy matters do not readily separate from the liquid in a distinct layer. In a solution, on the other hand, the dissolved substance is not merely broken up into small particles and held in suspension, but its identity becomes merged with that of the liquid solvent. It is to be noted that the greasy matters of the wool do not actually pass into solution but remain finely suspended as an emulsion, and in consequence of this may be separated from the scouring liquor by suitable mechanical treatment. The *suint*, on the other hand, passes into solution.

3. **Use of Alkali in Scouring.** — In adjusting the amount of *alkali* (soda ash) to be used in scouring, reference must be had to the quality and quantity of impurity in the wool, and also to the quality of the fibre. An excessive amount of alkali must be guarded against, as it is liable to injure the lustre and strength of the fibre, and also tends to discolor it. The finer the quality, of the fibre, as a rule, the less the amount of alkali that should be employed, and this is especially true of lustre-wools and fine merinos. Coarser and lower grade wools are scoured with a relatively larger amount of alkali. It is a mistake to presume that the more dirty and greasy the wool, the more alkaline should be the scouring liquors; this may be true when reference is had to wools of about the same quality, but a very dirty and greasy fine merino wool should be scoured with less alkali than a comparatively clean but low-grade Territory wool, on account of the greater liability to injure the fibre in the former case.

4. **The Scouring of Woolen Yarn.** — As the impurities in woolen yarn differ very materially from those present in raw wool, we would naturally expect a difference in the manner of scouring. The impurities in yarn, in the first place, are much less in amount, varying from 10 to 20 per cent. in ordinary woolen yarns, and from 2 to 5 per cent. with most worsted yarns. The character of

these impurities is also different; they consist of the oil which has been added during the spinning of the wool, together with the miscellaneous dust and dirt it may have collected passing through the various machines in carding and spinning. Oil is added to wool for spinning in order to make the fibres more plastic and to preserve them from mechanical injury. Such oil should be capable of easy removal from the spun yarn and should not add any deleterious substance to the wool. For instance, the oil should not be of a drying character, as it will form resinous products in the wool, the presence of which would be very disadvantageous; further, the oil should be capable of ready emulsion so that no difficulty may be experienced in scouring the yarn. Again, the oil should be free from acidity, as otherwise it would attack the card clothing and other metallic surfaces with which the wool may come in contact, not only causing injury to the machines but also causing the wool to become impregnated with iron compounds, which leads to many defects in subsequent scouring, bleaching, and dyeing. Owing to the fact that the impurities in yarn are of less amount and also more easily removed, it is customary to employ relatively less alkali in scouring than is the case with raw wool. Whereas, in scouring the latter substance the proportion of alkali is greater than that of soap, with yarn scouring the proportion is just reversed, and more soap than alkali is used; and furthermore the strength of the scouring liquors is much diminished. The exact composition of the scouring bath, also, in the case of yarn, must be regulated with reference to the amount of impurities in the fibre as well as the quality of the fibre itself. Worsted (and high-class yarns in general) containing but little oil and dirt are scoured in comparatively weak solutions containing a good quality soap and a minimum amount of alkali (or in some cases none at all). With lower grade and dirtier woollen yarns the proportion of alkali is increased. The temperature of the scouring bath for yarn, as with that for raw wool, is generally about 140° F., though with fine lustre-wools even this temperature is considerably reduced. Occasionally, with very low grade yarns, such as coarse carpet yarns (containing



a variety of crude hair fibres, such as goat and cow hair, coarse camel hair, mixed with jute and other vegetable fibres, as well as large quantities of inferior grease used in spinning), the temperature of the scouring liquor may be much increased, in some cases even as high as the boiling point.

**5. The Scouring of Yarns Containing Iron.** — In some cases woolen yarns are liable to contain quite appreciable quantities of iron, which may have been derived in a variety of ways, such as rusty cards, the use of an acid oil in spinning, contamination with the lubricating oil on the machines, etc. Such yarns are usually of a deep grayish color, and if scoured in solutions containing soda ash (or potash) will usually come from the scouring bath badly discolored by a yellowish brown stain. This is due to the iron becoming fixed in the fibre in the form of iron oxide (iron rust) by reaction with the alkali. Such yarn will exhibit serious defects in subsequent dyeing, as the iron will act as a "mordant" for many coloring-matters and tends to dull or "sadden" the color. Yarn of this character should be scoured in solutions containing only soap without the addition of any alkali, so as to permit of the proper removal of the iron rather than its fixation in the fibre.

**6. Soaps for Scouring Wool.** — A soap is a combination between an alkali and a fatty acid, and is produced by the action of a caustic alkali on an oil or fat. The latter substances (whether of vegetable or animal origin) are compounds of glycerin with various fatty acids, and by proper treatment with caustic soda or caustic potash are decomposed with the liberation of glycerin and the formation of a soap. Caustic soda yields *hard* soaps, whereas caustic potash gives *soft* soaps. The different oils and fats, naturally, furnish soaps of different characteristics, and some soaps are more suitable for scouring than others. A good scouring soap should be readily soluble in water and possess high emulsifying powers towards greasy matters; it should contain no fats which would act deleteriously on the fibre or leave it with an objectionable odor or color. The soap should furthermore be capable of easy removal from the wool after scouring, and not

leave behind any resinous or fatty matters of an insoluble character. As already mentioned, it should also not contain any appreciable quantity of free caustic alkali; nor, on the other hand, should it contain unsaponified fat. Soaps made from olive oil are usually considered of the highest grade and the most desirable for wool scouring; although soaps made from cotton-seed oil, maize oil, tallow, oleine (the liquid fat obtained as a by-product in candle-making), and palm oil are also extensively employed. Often mixed soaps are used, such as olive or cotton-seed oil soap in combination with a tallow soap. Both hard and soft soaps are used, though the latter are generally preferred for scouring raw wool as well as yarn.

**7. Relation of Water to Wool Scouring.** — The use of the proper kind of water in wool scouring, both for the preparation of the scouring bath and for the washing of the wool after scouring, is a matter of considerable importance. The use of *hard* water, as such, should be interdicted, on account of its bad action with soap solutions. By a “hard” water is meant one containing considerable amounts of dissolved mineral substances, usually compounds of calcium (lime), magnesium, and sometimes iron. These mineral substances in solution combine with soap to form insoluble and sticky precipitates, which cause not only a loss of soap and a consequent decrease in the scouring power of the bath, but also these precipitates adhere to the fibre and are difficult of removal. Sometimes the “hardness” of water is removable by simply boiling; it is then termed *temporary hardness*, and is principally due to the presence of carbonic acid gas holding calcium carbonate (limestone) in solution. On boiling, the carbonic acid gas is driven off and in consequence the calcium carbonate is precipitated. *Permanent hardness*, on the other hand, is not removable by boiling, and is chiefly due to the presence of calcium sulphate (gypsum). To remove hardness of this character soda ash is usually added sufficient to precipitate the calcium compound as the highly insoluble carbonate. Or, small quantities of oxalic acid and ammonia may be added, which causes the precipitation of the lime as calcium oxalate.

Before hard water is used in connection with wool scouring it should be "corrected" in one of the ways here indicated. Water containing any appreciable amount of iron is especially objectionable for use in scouring, as the iron readily becomes firmly fixed in the fibre, leading to many bad defects. Iron may be best removed from water by proper aëration and filtration. Should the water contain sediment in any appreciable amount it should be properly filtered before use. River or pond water is liable to contain a larger amount of sediment than spring or well water, but, on the other hand, as a rule, its hardness is not so great, nor is it as liable to contain iron.

**8. Boiling-out of Cotton.** — Raw cotton is unlike wool in that it contains a relatively small amount of natural impurity, and for many purposes cotton is not scoured at all. Whereas raw wool cannot be manufactured into yarn without a previous removal of its greasy and dirty matters, cotton is spun without any such preliminary cleansing being required; in fact the impurity that is present in raw cotton is an aid rather than a hindrance to the proper spinning of this fibre. In many cases of dyeing, also, a previous scouring of cotton is not required. The impurities in raw cotton consist for the most part of waxy and resinous matters, which are classified under the general term of *pectin* substances. These amount to about 5 per cent. on the weight of the fibre. By reason of its coating of waxy matters, the cotton fibre is more or less waterproof, or rather water-repellent, and will not readily "wet-out" when placed in water. This property is frequently a drawback in dyeing, as the dye solution will not penetrate perfectly and evenly. To overcome this defect it is necessary to remove the waxy coating on the cotton, and this is best done by boiling in a solution of caustic soda, soda ash, or soap, or with some oil which has the property of dissolving the cotton-wax. This "wetting-out" of cotton for purposes of dyeing or mordanting is simply with a view of allowing it to become quickly and thoroughly saturated with the solutions in which it may be placed. When cotton is to be bleached, however, it is not only necessary to scour the fibre



so that it will readily wet-out, but also to completely remove all resinous substances, otherwise a good clear white will not be obtained in the bleaching. For this purpose, it is generally necessary to boil the cotton with a solution of caustic soda (or other alkalis) for a number of hours and usually under more or less pressure. This operation is termed "boiling-out" to distinguish from mere "wetting-out." In the scouring of cotton it is probable that the waxy and resinous substances in the fibre are emulsified or dissolved. Caustic soda is probably the most generally employed chemical for the scouring of cotton, though soda ash and sodium silicate are also extensively used. Often mixtures of these three are employed. Soap is also an efficient medium for wetting-out cotton, though it appears that when a very thorough boiling-out process is required a more strongly alkaline agent is desirable. Formerly lime[(slaked in water)] was extensively employed for boiling-out cotton preliminary to bleaching, but its use is rapidly giving way to that of caustic soda. Certain so-called "soluble oils" (prepared by treating castor oil, cotton-seed oil, etc., with strong sulphuric acid, and hence also called "sulphated" oils) appear to possess the property of quickly dissolving the waxy matters from cotton, and these are sometimes used for the purpose of wetting-out of cotton for dyeing. They are also at times used in the process of boiling-out. Fankhausine, Solvine, Monopol Oil, etc., are compounds of this character, and consist, for the most part, of sulphated vegetable oils. For the purpose of merely wetting-out it is probably better to use either a solution of soap or a soluble oil, rather than the alkalis, as the former method leaves the cotton somewhat whiter in appearance and softer in feel. It is probable that when boiled with solutions of caustic soda or soda ash the resinous matters in the fibre are decomposed with the formation of brown coloring-matters, and as a result the cotton has a darker color than when treated with soap or oil.

**9. The Impurities in Raw Silk.** — Raw silk, as it appears in trade, does not much resemble the brilliant and lustrous fibre seen in manufactured silk fabrics. Raw silk consists not only

of the fibre proper, but also of a large amount of a glue-like substance which heavily coats the fibre and gives it a harsh, brittle feel and hides the lustre and whiteness of the true fibre. This substance is known as *silk-glue* or *sericin*, and it amounts to about 25 per cent. of the weight of the raw silk. It is soluble in water, and may indeed be completely removed from the fibre by prolonged boiling. It is, however, more readily removed by strong solutions of soap, and this is the usual method employed. The fibre proper of silk is known as *fibroin*, and though very similar in chemical composition to sericin, it is insoluble in water or soap solutions. The most of the coloring-matter in raw silk is also contained in the silk-glue and is removed along with this latter substance. Certain raw silks (yellow-gum Italian for instance) are of a deep yellow color, but when completely stripped of silk-glue they become as white as other silks.

**10. The Boiling-off of Silk.** — The scouring of raw silk, or the removal from it of the silk-glue, is usually termed “boiling-off,” though the expressions “degumming” and “stripping” are also used. When completely boiled-off silk will lose in weight from 22 to 28 per cent. Frequently, however, all of the silk-glue is not removed, but only sufficient to make it soft and lustrous and workable in dyeing or bleaching. Under these circumstances, the scouring of silk is termed *soupling*, and only from 10 to 15 per cent. in weight is lost. Furthermore, raw silk is sometimes given only a very slight scouring for the purpose of softening the fibre; this gives what is called *écru* silk, and only 2 to 5 per cent. in weight of the silk-glue is removed. The scouring of silk is almost invariably accomplished by the use of boiling solutions of soap. The length of time and the number of soapings given will determine how much of the sericin will be removed. For a complete boiling-off a strong soap solution is necessary (about one pound of soap per gallon), and the time required is from 1 to 2 hours, and this treatment is usually repeated with a second soap solution. The soap employed for the scouring of silk should be of the very best quality, and should be as neutral as possible. The presence of any appreciable

free alkali in the scouring bath will rapidly injure the silk fibre, causing it to become weakened, discolored, and lustreless. Generally, the best grade of hard olive oil soap is used; soft soaps are not employed because these are nearly always liable to contain small quantities of free alkali. The scouring baths left after the boiling-off of silk (and usually repeated lots of raw silk are scoured in the same soap solution) contain a large quantity of silk-glue together with the soap employed. These residual baths are known as *boiled-off liquors* and are extensively used as an adjunct in the dyeing of silk, being added in considerable amount to the dye-bath for the purpose of softening the dyed silk and promoting the even distribution of the color. After the silk has been scoured or boiled-off it should be thoroughly washed with water in order to remove all trace of soapy liquor, otherwise the soap will dry into the fibre and cause discolorations and imperfections. After the scoured silk is dried, in order to soften the fibre and to give it increased lustre, it is stretched or steamed. This is merely a mechanical treatment which loosens up the fine and delicate filaments of the silk fibre which have become more or less matted together in the scouring and drying.

#### SAMPLES.

22. Raw wool in the grease.
23. Wool scoured with soap and soda ash.
24. Wool scoured with soap and potash.
25. Wool scoured at a boiling temperature.
26. Wool scoured with excessive alkali.
27. Woolen yarn in the grease.
28. Woolen yarn scoured.
29. Woolen yarn containing iron in the grease.
30. Woolen yarn containing iron scoured with soda ash.
31. Woolen yarn containing iron scoured with soap alone.
32. Cotton yarn; unscoured.
33. Cotton yarn scoured with caustic soda.
34. Cotton yarn scoured with soda ash.
35. Cotton yarn scoured with soap.
36. Cotton yarn scoured with Fankhausine.
37. Raw silk in the gum.
38. Boiled-off silk.
39. Boiled-off silk after stretching.



## RECORD OF RESULTS.

Test.	Weight before scouring.	Weight after scouring.	Loss in weight.	Shrinkage. Per cent.
6.....				
7.....				
8.....				
9.....				
10.....				
12.....				
13.....				
14.....				
15.....				
16.....				

## QUIZ 2.

27. Describe the general character of the different impurities existing in raw wool.

28. How does wool grease differ from other animal fats in its chemical composition? How does wool grease react with water and alkaline or soap solutions?

29. What is suint and of what does it consist? Is it soluble or insoluble in water?

30. What chemicals are employed in the usual method of wool scouring? At what temperature is the scouring operation conducted?

31. Why is it necessary that the soap and alkali employed in wool scouring should be as free as possible from caustic alkali?

32. Why is it necessary to wash the wool thoroughly after scouring, and why is warm water employed?

33. What percentage of impurities is present in raw wool? Is this amount at all constant for different wools? How does the amount of grease in fine wools compare with that present in coarse wools?

34. What is meant by the shrinkage of raw wool? Why is it important for the wool buyer to know this factor?

35. Based on the quantities given in the test, how many pounds of soap and soda ash would be required to prepare a scouring bath containing 1000 gallons of water?

36. What is potash? Why is its use for wool scouring recommended in place of soda ash? How did your results in scouring with the two compare?

37. Why is wool not scoured at temperatures above 140° F.? What is the effect of scouring at the boil? How does the sample of wool scoured at the boil compare with samples scoured at lower temperatures?

38. What is the effect of employing an excessive amount of alkali in the scouring bath?

39. How does the general method of scouring woolen yarn differ from that of scouring raw wool with respect to the relative proportions of soap and alkali?

40. Describe the general nature of the impurities in woolen yarn. Why is wool oiled in spinning, and what should be the character of the oils employed?

41. Why is some woolen yarn liable to contain iron in the grease? How should yarn of this character be scoured? Why should soda ash not be employed?

42. What is the general nature of the impurities existing in raw cotton? What is their average amount?

43. Why is it necessary to scour cotton before dyeing or bleaching? Is a previous scouring always required before dyeing?

44. How does the "wetting-out" differ from the "boiling-out" of cotton?

45. What is the general principle involved in the scouring of cotton? What chemicals may be employed for this purpose?

46. Give a comparison of the results obtained in the boiling-out of cotton with caustic soda, soda ash, and soap. About how much does cotton lose in weight by boiling-out?

47. What is Fankhausine? How does it act in the scouring of cotton? How does cotton boiled-out with Fankhausine compare with that boiled-out with alkalies?

48. What substances are present in raw silk? In what relative amounts do these exist?

49. How does sericin differ from fibroin? How does raw silk differ in its general appearance from boiled-off silk?

50. What substance is removed in the scouring of silk? By what different names is the scouring of silk known? How much does silk lose in weight by scouring?

51. How is the boiling-off of silk conducted? What chemicals are employed and at what temperature?

52. What kind of soap should be employed for boiling-off silk? Why should the soap be as neutral as possible?

53. What strength of soap solution is employed for boiling-off silk? On the basis of the figures given in the experiment, how many pounds of soap would be required to make up 100 gallons of liquor for scouring silk?

54. What is the difference between "écru," "soupled," and "boiled-off" silk?

55. Does the complete removal of the silk gum from raw silk generally take place in a single scouring bath?

56. How is the boiled-off silk softened and lusted? In what constituent of raw silk does the natural coloring-matter principally occur, and is it removed in the boiling-off?

57. What is meant by "boiled-off liquor"? Of what does it consist? For what purpose is it employed?

### SECTION III.

#### BLEACHING OF WOOL.

**Experiment 17. Bleaching Wool by Tinting.** — Take a well-scoured test-skein of woolen yarn (40), and work in a lukewarm bath containing a trace of oxalic acid and a trace of Acid Violet 2 B (about  $\frac{1}{100}$  per cent. on the weight of the wool will as a rule be ample dyestuff). Take great care not to add too much of the coloring-matter, otherwise too distinct a color will be imparted to the wool. After tinting, squeeze and dry (41). It will be found that the violet coloring-matter has neutralized the yellowish tint of the wool, so that the material seems whiter than before. To show the same operation on cotton, take a test-skein of cotton yarn (42) which has been well scoured out with 2 per cent. of Monopol Soap, and work it in a dilute lukewarm soap bath containing a trace (about  $\frac{1}{100}$  per cent. on the weight of the cotton) of Methyl Violet 5 B. Then squeeze and dry (43). It will be found that, as with the wool, the skein of cotton will appear whiter after tinting, owing to the fact that the violet-blue color has destroyed the yellowish color of the natural fibre.

**Experiment 18. Bleaching Wool with Sulphurous Acid Gas.** — Take a skein of well-scoured woolen yarn, wet it out in water, then squeeze it so that the wool is left only moist; place it in a compartment filled with sulphurous acid gas for 12 to 24 hours. Then wash well in water, and then in a bath containing a trace of oxalic acid and Acid Violet for tinting (44).

**Experiment 19. Bleaching Wool with Sodium Bisulphite.** — Prepare a bath containing 300 cc. of water and 10 cc. of sodium bisulphite solution (32° Tw.). Immerse two well-scoured skeins of woolen yarn in this bath, work well for about 15 minutes, then allow to soak for 12 to 24 hours. Then squeeze and work in a bath containing 5 per cent. of sulphuric acid (on the weight



of the wool). Then wash the first skein well in water, and finally in a bath containing a trace of oxalic acid and Acid Violet for tinting. Then squeeze and dry (45). Take the second skein so bleached and pass through a cold bath containing a couple of drops of a dilute solution of potassium permanganate (just sufficient to give the water a violet color), and then wash again (46). If too strong a solution of the potassium permanganate is used the wool will acquire a brownish color, and will have to be passed through a dilute bath of sodium bisulphite in order to remove the brown hydrate of manganese which will be precipitated on the fibre. Cut about 6 inches from each of the two bleached skeins and plait with portions of a skein of woolen yarn which has been dyed with Magenta (a dyestuff quite susceptible to the action of sulphurous acid), and allow the samples thus prepared to remain for several days. The skein of dyed yarn may easily be prepared by working a skein of woolen yarn in a bath containing 300 cc. of water and about 5 cc. of a solution of Magenta for one-half hour at a temperature of 180° F. On examination after a time it will be found that the bleached skein which was not treated with the potassium permanganate solution has caused a discoloration of the dyed sample with which it was plaited (47), whereas the other bleached skein has not (48). This test shows the presence of sulphurous acid in the former and the absence of it in the latter.

**Experiment 20. Bleaching Wool with Sodium Peroxide.** — Prepare a bath containing 300 cc. of water and 3 cc. of concentrated sulphuric acid; then carefully add with constant stirring, 4 grams of sodium peroxide. Test with litmus paper, and if not acid in reaction, add sufficient dilute sulphuric acid to turn the paper red. This will neutralize all of the caustic soda formed in the decomposition of the sodium peroxide with the water. Now add sufficient sodium silicate solution to make the bath slightly alkaline; that is, until it turns the litmus paper blue again. Heat the bath to 120° F., when it is ready for bleaching. Take a well-scoured skein of woolen yarn and work it in this bath for 15 minutes, and then allow it to steep under the liquor for 12 to 15

hours, maintaining the temperature as nearly as possible at about 100° F. during that time. Then wash well and squeeze, and finally tint in a bath containing a trace of oxalic acid and Acid Violet. Then squeeze and dry (49).

**Experiment 21. Bleaching Wool with Potassium Permanganate.** — Prepare a bath containing 300 cc. of water and 0.2 gram of potassium permanganate and 5 per cent. (on the weight of the wool) of sulphuric acid. Warm the bath to 100° F., and steep a well-scoured skein of woolen yarn therein for about 5 minutes, working during that time. Then rinse, and it will be found that the wool has become brown in color (50); this is due to the precipitation of hydrated oxide of manganese on the fibre, resulting from the decomposition of the potassium permanganate. Next work the skein in a cold bath containing 300 cc. of water and 2 cc. of sodium bisulphite solution (of 32° Tw.). The wool will rapidly turn white (51), as the brown deposit of manganese oxide is dissolved by the bisulphite of soda.

#### NOTES.

**1. Bleaching Wool.** — The wool fibre in its natural condition always contains some pigment matter; even the usual so-called "white" wool contains a small amount of a yellowish brown color which it is necessary to remove in order to have a fibre possessing a clear white color. In some grades of wool the amount of pigment matter may be comparatively large, giving the brown or black wools. These wools, however, are small in amount compared with the white wools and are seldom, if ever, bleached. The method of bleaching wool by the tinting process described in Exp. 17 depends on the neutralization of the slight yellow tint of the natural wool by dyeing the fibre with a delicate tint of blue or violet coloring-matter. It is not really a removal or destruction of the natural pigment, but simply a change of the yellow tint to one of a grayish tone. The latter being less susceptible to the eye causes the wool to appear white. The color relations in the case are based on the fact that yellow and violet are complementary colors, so that when mixed in small amount

they produce gray. For the tinting color it is best to use a blue dyestuff with a slight violet tone, such as a very blue tone of Acid Violet. Oxalic acid is used with the dyestuff to render the solution slightly acid and thus develop the color. The actual amount of color required is very small and care must be exercised not to overtint the wool, or a bluish tone will be obtained. Wool bleached in this manner, of course, will not possess as clear a white color as that in which the natural pigment is actually destroyed; it will only give a dull, cloudy-looking white.

Sulphurous acid, or one of its compounds, is the agent mostly employed for the true bleaching of wool. Sulphurous acid is a strong reducing agent; that is to say, it has a strong "affinity" for oxygen. When acting on many organic coloring-matters (such as the natural pigment in wool) it "reduces" them, thus causing them to be converted into colorless substances. Many coloring-matters, however, after being thus reduced, are capable of becoming oxidized on exposure to air so as to yield again the original color; this appears to be the case with the coloring-matter in wool, for when bleached with sulphurous acid the yellow tint becomes gradually restored on exposure to the air.

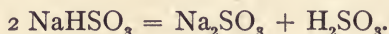
Bleaching by the use of sulphurous acid gas is the method mostly practiced for the bleaching of wool. The process is rather simple; the wool (either in loose state, yarn, or cloth) is moistened and spread out or hung in a room where it is subjected to the action of the sulphurous acid gas for 10 to 20 hours. The gas is produced generally by the burning of sulphur in an iron or earthenware pot, sometimes in the bleaching room itself, though it is considered better to burn the sulphur in an apparatus outside the bleaching room and to lead the gas into the latter. From the use of the so-called "stove" for burning the sulphur, this process of bleaching has received the name of "stoving." The wool (in whatever form) must be thoroughly scoured for bleaching and should be in a moist (though not *wet*) condition, as the gas acts but slowly on the dry wool. The material should also be so distributed in the bleaching room that the gas may easily come in contact with all parts of the fibre. Usually the





gas is allowed to pass from one end of the room to the other and thence out through a flue. The bleaching chamber must be so constructed that the condensed vapors (which consist of rather concentrated sulphuric acid) cannot drop on the wool, else spotting will result, or the fibre may even be seriously injured. Also the room should not contain exposed iron parts which may come in contact with the sulphur gas, as the metal will rapidly be attacked and the condensed drops that may fall on the wool will cause bad spotting.

**2. Use of Sodium Bisulphite.** — The use of this chemical for the bleaching of wool is merely a convenient method for the application of sulphurous acid in the form of a solution. The bleaching agent, in fact, is exactly the same as when sulphurous acid gas is employed, and the character of the bleach obtained in the two cases is practically identical. Sodium bisulphite has the chemical formula  $\text{NaHSO}_3$ , and when dissolved in water its solution practically consists of sodium sulphite and sulphurous acid:



When wool is steeped in this solution the sulphurous acid acts directly upon the fibre as a bleaching agent, and moreover, the wool also becomes saturated with the sodium sulphite. Hence the wool is subsequently treated with a solution of sulphuric acid, which reacts with the sodium sulphite, forming sodium sulphate and liberating another portion of sulphurous acid:



This second portion of sulphurous acid also aids materially in the bleaching of the wool.

Bleached wool is usually tinted with a blue or bluish violet coloring-matter in order to give to the fibre a bluish white tone which is more pleasing to the eye than the flat bleach. A minute quantity of a blue shade of Acid Violet is useful for this purpose, and it is generally applied in the rinsing bath after the bleaching, adding a small quantity of oxalic acid to the water for the purpose

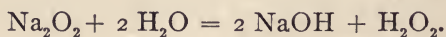


of developing the color and also for the purpose of removing any trace of brownish stain due to the presence of iron compounds.

Mention has already been made of the fact that the bleach obtained on wool by means of sulphurous acid is not a permanent one, but the yellow tint reappears after prolonged exposure to the air. Furthermore, it appears to be practically impossible to remove every trace of sulphurous acid from the fibre, however thorough the washing may be after the bleaching. The wool apparently combines in a chemical manner with the sulphurous acid, and this leads to two defects in the bleached wool; in the first place, the presence of the sulphurous acid apparently holds the pigment in the fibre in a reduced state so that the bleach lacks permanency of character, as already noted; secondly, the presence of the sulphurous acid is liable to act injuriously on other dyed colors with which the bleached wool may subsequently come in contact when woven into cloth. This effect is illustrated in the experiment by the action of the bleached wool in contact with wool dyed with Magenta. For these reasons it has long been recognized as desirable to remove from the bleached wool all trace of sulphurous acid. This may readily be accomplished by treating the bleached material with a solution containing a suitable oxidizing agent. Potassium permanganate has been quite extensively employed for this purpose. By its action the sulphurous acid is converted into sulphuric acid, which is harmless as far as the effects outlined above are concerned. In the use of this agent, however, great care must be exercised not to employ an excess beyond that needed to react with the sulphurous acid; otherwise a brown deposit of an oxide (or hydrate) of manganese will be left on the wool, and a subsequent treatment with a solution of sodium bisulphite will have to be given to remove this deposit. Instead of using potassium permanganate in this connection it would probably be better to employ a small quantity of sodium peroxide, which would have the same effect on the trace of sulphurous acid without the attendant defect of discoloration through the addition of an excess of the reagent. The presence of

traces of sulphurous acid in wool may be conveniently detected by wetting the wool in a small quantity of water and adding a few drops of a mixture of iodic acid and starch solutions; if sulphurous acid is present a violet or blue color will be formed.

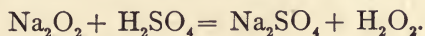
**3. Bleaching Wool with Peroxides.** — The use of sodium peroxide as a bleaching agent for wool is fast becoming of considerable practical importance. Formerly hydrogen peroxide was somewhat employed for this purpose, but its high cost was a bar to its extensive application. The bleaching action of these two substances, however, is identical, and is due to the nascent oxygen which they are capable of liberating. Hydrogen peroxide has the chemical formula  $H_2O_2$ , and is prepared by the action of sulphuric acid on barium peroxide. As employed in the arts it consists of a comparatively dilute solution (about 3 per cent.) of hydrogen peroxide in water. The chemical formula of sodium peroxide is  $Na_2O_2$ ; it is prepared by heating metallic sodium in air. It occurs as a yellowish white powder and may be obtained of a high degree of purity. Some care must be taken in the handling and using of sodium peroxide, as it is easily decomposed in the presence of moisture and organic matter with the evolution of large volumes of oxygen which may lead to explosions or fires. When handled with intelligent precaution, however, it is by no means a dangerous chemical. It should be stored in a cool, dry place in comparatively small tins (the usual commercial size is that containing 10 pounds), and should be kept from contact with water or with organic matter such as paper, excelsior, etc. As the reaction which occurs between sodium peroxide and water is a very violent one, its solution should be carefully undertaken. *Large quantities or lumps of sodium peroxide should never be added to water*, as an explosion or fire is liable to result. The peroxide should be sifted gradually into the water with constant stirring. When sodium peroxide is dissolved in water caustic soda and hydrogen peroxide are formed:



Its bleaching effect is due to the ready decomposition of the hydrogen peroxide in contact with organic matter (such as wool):



The oxygen, at the moment of its liberation in such a manner, is especially reactive (so-called *nascent* oxygen), and easily destroys the organic coloring-matters of which the pigment of the wool consists. It is necessary to neutralize the caustic soda in the solution by the addition of sulphuric acid, as the presence of the caustic alkali in the bleaching bath would rapidly destroy the wool fibre. On this account the bath is usually prepared by first adding the requisite amount of sulphuric acid to the water, and then slowly adding the sodium peroxide. Under these circumstances the peroxide reacts with the sulphuric acid to form sodium sulphate (glaubersalt) and hydrogen peroxide:



In order to insure the fact that there is no free caustic soda in the solution it is best to use a slight excess of acid, which may be indicated by testing the bath with a piece of blue litmus paper. This will be turned red in the presence of an excess of acid. The bleaching effect of the dissolved hydrogen peroxide, however, is stronger in an alkaline solution than in an acid one; this is due to the fact that the peroxide more readily decomposes in the former solution. Therefore where the bleaching bath is in actual use it should be made slightly alkaline with a reagent which will not be injurious to the wool. Sodium silicate has been found to be most suitable for this purpose, though ammonia or borax may also be used. In this connection it must be remarked that a large excess of sulphuric acid must be avoided, otherwise when the silicate is added it may separate in a jelly-like mass and ruin the bath. During the bleaching of the wool the bath should be maintained at a temperature of about 100° F. If the temperature is much higher than this the hydrogen peroxide will be too rapidly decomposed and loss of oxygen will be occasioned; if the bath is too strongly alkaline a similar condition will result.



The sodium peroxide bleaching bath must be contained in a wooden vat and the pipes used for connections and heating should be of lead. The presence of all other metals, especially iron, should be rigidly excluded; even the sulphuric acid and the water employed in the bath should be perfectly free from iron, otherwise very inferior results will be obtained. A suitable strength for the bleaching bath is 5 lbs. 5 ozs. of sulphuric acid (168° Tw.) and 4 lbs. of sodium peroxide (98 per cent.) per 100 gallons of water. The character of the wool or the nature of the material to be bleached may necessitate a somewhat stronger bath than this, in which case the same relative proportions of acid and peroxide should be used.

Wool bleached with sodium peroxide does not exhibit the same defects as noted under the bleaching with sulphurous acid. It does not retain any substance deleterious to dyed colors, nor does the yellow tint of the natural pigment return on exposure to the air, for this pigment appears to be permanently destroyed by the peroxide. Attention may here be drawn to the radical difference in the principle of bleaching with sulphurous acid and with sodium peroxide. In the former case the bleaching takes place through the *reducing* action of the sulphur dioxide, whereas in the latter case the bleaching is brought about by the strong *oxidizing* action of the peroxide.

In order to ascertain if the bleaching bath of sodium peroxide after use still contains active oxygen for further use in bleaching the following test may be carried out: A small quantity of the residual liquor is placed in a test-tube and a few drops of a dilute solution of potassium permanganate are added. If the bath still possesses an oxidizing action, the violet color of the permanganate solution will be destroyed.

**4. Bleaching Wool with Potassium Permanganate.** — This compound is also a strong oxidizing agent, and its solution will rapidly destroy the natural pigment of wool. In the decomposition of the permanganate, however, whereby it liberates oxygen, there is also formed an insoluble hydrated oxide of manganese, which is precipitated in the wool and imparts to it a brown color.



The decomposition (or oxidizing action) of the permanganate is facilitated by the presence of sulphuric acid, and the bleaching effect is completed in a relatively short space of time. In order to remove the insoluble brown compound of manganese from the fibre it is best to treat the material in a cold dilute solution of sodium bisulphite. The sulphurous acid present in the latter solution reacts with the manganese compound to form a colorless soluble product, and the fibre is left in a clear white condition. Care must be taken in this connection not to employ an excess of sodium bisulphite solution, otherwise sulphurous acid will be left in the wool, and will exhibit the defect already noted under the consideration of the sulphurous acid bleach. If this latter defect is avoided, the permanganate bleach on wool is probably as satisfactory as the peroxide bleach. It can also be carried out in much less time. Too strong a solution of permanganate must be avoided, otherwise the wool will acquire a harsh feel, due to the oxidation of the fibre.

With regard to the comparative cost of the several methods of bleaching wool, it may be stated, in general, that the sulphurous acid bleach is the cheapest, while the peroxide method is the dearest. An approximation to the cost of the three methods (for yarn) is as follows:

Sulphur bleach (gas) .....	1 $\frac{3}{4}$ cts. per lb.
Permanganate bleach .....	2 $\frac{1}{2}$ cts. per lb.
Peroxide bleach .....	4 $\frac{1}{2}$ cts. per lb.

The permanganate method has not come into favor as yet, apparently on account of its being more difficult to regulate.

#### SAMPLES.

40. Woolen yarn before bleaching for comparison.
41. Woolen yarn bleached by tinting process.
42. Cotton yarn before bleaching for comparison.
43. Cotton yarn bleached by tinting process.
44. Woolen yarn bleached with sulphurous acid gas.
45. Woolen yarn bleached with sodium bisulphite.
46. Sodium bisulphite bleach treated with permanganate.

47. Effect of sulphurous acid in yarn on dyed colors.
48. Effect on dyed colors after removing sulphurous acid.
49. Woolen yarn bleached with sodium peroxide.
50. Woolen yarn treated with potassium permanganate.
51. Woolen yarn bleached with permanganate and bisulphite.

### QUIZ 3.

58. What is the natural color of ordinary wool? Explain the theory of bleaching by means of the tinting process. What coloring-matters are employed, and how is the operation conducted?

59. How may the bleaching action of sulphurous acid on organic coloring-matters be explained?

60. Give the process of bleaching wool with sulphurous acid gas. What length of time is required? Why is it necessary to tint the bleached material? What is meant by "stoving"?

61. What is sodium bisulphite? What is its active bleaching principle?

62. Give the complete process of bleaching wool with solutions of sodium bisulphite.

63. Explain the use of the bath of sulphuric acid in bleaching with sodium bisulphite.

64. What disadvantages are attached to the sulphurous acid method of bleaching wool? How could you test the bleached wool to show if it contained sulphurous acid?

65. How may the sulphurous acid left in the wool after bleaching be removed or neutralized?

66. What defect is liable to arise when yarn bleached with sulphurous acid is woven with dyed yarns?

67. What difference, if any, did you notice in the samples prepared from the treated and untreated bleached skeins when plaited with a dyed yarn?

68. What is the active bleaching principle in peroxides? What is hydrogen peroxide? How is it made? Why is it not much used directly for bleaching purposes?

69. What is sodium peroxide? How is it made? What precautions must be taken in handling and using it?

70. What reaction takes place when sodium peroxide is dissolved in water? What reaction takes place when it is dissolved in dilute sulphuric acid?

71. Give the method of preparing the bleaching bath with sodium peroxide. Why should the bath still be slightly acid after the peroxide is added?

72. What is the purpose of making the peroxide bleaching bath slightly alkaline before bleaching, and what alkalies may be employed for the purpose?

73. At what temperature is the bath maintained in bleaching with sodium peroxide? What length of time is required for the bleaching?

74. Describe a test which will indicate whether the peroxide bath is exhausted or not. If the peroxide bath is to be kept over how is it best preserved? In what kind of a vat should the peroxide bleaching bath be used? How is the bath heated?

75. How does the quality of the peroxide bleach on wool compare with that with sulphurous acid in regard to cost, permanency, and action of dyed colors woven with the bleached yarn?

76. What is the effect of traces of iron in the peroxide bleaching bath? Do other metals have the same effect? Why should all the chemicals employed in the bath be perfectly free from iron?

77. What is potassium permanganate? What is its active bleaching principle?

78. How is the permanganate bleaching bath prepared? Explain the chemical reaction which occurs when wool is worked in an acidulated solution of potassium permanganate.

79. What is the appearance of the wool after coming from the permanganate bath? To what is this due?

80. What after-treatment is necessary in the permanganate bleach? Explain the chemical reaction which takes place.

81. At what temperature is the bleaching with potassium permanganate conducted? What time is required for the bleaching? How does the result compare with the other methods of bleaching?

## SECTION IV.

### BLEACHING OF COTTON.

#### **Experiment 22. Bleaching Cotton with Chloride of Lime. —**

Take a weighed skein of cotton yarn and boil it out in caustic soda as described in Exp. 12. Take another weighed skein and boil it out with soap as described in Exp. 14; also a third weighed skein scoured with Monopol Soap as in Exp. 15. Wash these skeins well and steep in a solution of chloride of lime (bleaching powder) of 2° Tw. strength. Work for several minutes until the fibre is thoroughly saturated with the liquor; then immerse in the solution and allow to stand for 1 hour. Then squeeze and rinse in water, and "sour" by passing for 15 minutes through a cold bath of sulphuric acid of 1° Tw. strength. Finally wash well in water to remove all trace of acid. Reweigh each skein (52, 53, 54) and calculate the percentage of loss in each case, and compare the bleach obtained with each method of boiling-out. Test the skeins for *acid* by moistening a portion with a little water and pressing against it a piece of blue litmus paper; if the test paper turns red, acid is still present in the fibre, and the washing has been imperfect, with a result that the cotton will soon become tender. To test the bleached cotton for traces of *chlorine* which may remain after bleaching, take a portion of the skein and warm with a small amount of potassium iodide-starch solution; if a blue color is developed, there is still chlorine in the fibre. The test solution may be prepared by dissolving a little starch paste (made by boiling up some starch with water to a paste) in a solution of potassium iodide.

**Experiment 23. Use of "Anti-chlor" for Removing Chlorine in Bleaching. —** Take a skein of cotton yarn which has been boiled out in caustic soda in the usual manner, and bleach it for



1 hour in a cold solution of chloride of lime at 2° Tw., then wash in water. On a portion of the skein place a drop or two of the potassium iodide-starch solution, and it will be found that a blue color is developed, showing the presence of chlorine. Now pass the skein through a bath containing a little sulphuric acid for a few minutes, and wash again. Test with the potassium iodide-starch solution again, and it will still be found that free chlorine is present. Prepare a bath containing 300 cc. of water and 1 gram of sodium thiosulphate (sodium hyposulphite) and pass the skein through this solution cold for 10 minutes. Wash (55) and again test with the potassium iodide-starch solution, when it will be found that the free chlorine has been neutralized. The sodium hyposulphite is called "anti-chlor" when used for this purpose; sodium bisulphite will also answer the same purpose.

**Experiment 24. Bleaching Loose Cotton for Absorbent Purposes.** — Weigh out 10 grams of loose cotton (56) and boil for 1 hour in a bath containing 300 cc. of water and 5 grams of caustic soda. Rinse off well in fresh water and bleach in a cold solution of chloride of lime at 2° Tw. for 1 hour. Rinse, and pass through a cold solution of sulphuric acid at 1° Tw. for 20 minutes. Then wash well in several changes of water until all acid is removed. Then squeeze and dry (57). Reweigh the sample and calculate the percentage of loss. Test the bleached cotton for absorbent qualities by placing a small bit of it on the surface of cold water; if it is perfectly absorbent, it should sink at once. Try a small piece of raw cotton in the same manner, and it will be found that the latter does not sink at all.

**Experiment 25. Tinting and Softening of Bleached Cotton.** — Boil out four skeins of cotton yarn in caustic soda in the usual manner; wash well in water, and bleach for 1 hour in a cold bath of chloride of lime at 2° Tw. Wash, squeeze, and pass for 15 minutes through a cold bath of sulphuric acid at 1° Tw. Wash in two changes of water. Then test the skeins with litmus paper, and the chances are that they will still show the presence of acid. Set one of the skeins aside for comparison (58). Take another one of the skeins and work for 15 minutes in a bath

containing 300 cc. of water, 1 gram of soap, and  $\frac{1}{100}$  per cent. of Methyl Violet 5 B; have the temperature at about 140° F. Then squeeze and dry (59). Take a third skein and treat in the same soap bath, but add  $\frac{1}{100}$  per cent. of the coloring-matter; squeeze and dry (60). Take the fourth skein and treat in the same manner with the soap solution, but add  $\frac{1}{50}$  per cent. of the dyestuff; squeeze and dry (61). The percentages of the dyestuff are to be calculated on the weight of the cotton tinted. That is,  $\frac{1}{100}$  per cent. on 10 grams of cotton would be 0.0005 gram. The solutions provided should contain 0.1 gram of dyestuff per litre (1000 cc.); hence 1 cc. would represent 0.0001 gram of dyestuff, and it would require 5 cc. to give the necessary  $\frac{1}{100}$  per cent., or 10 cc. for  $\frac{1}{50}$  per cent., or 20 cc. for  $\frac{1}{25}$  per cent. After the several skeins have dried, compare the feel or softness of the first with that of the others which have been treated with the soap bath. Also compare the degrees of tinting of the latter three skeins and the difference in the character of the white obtained in each case.

**Experiment 26. Use of Acetic Acid in Bleaching.** — Boil out a skein of cotton yarn with caustic soda in the usual manner and bleach for 1 hour in a cold solution of chloride of lime at 2° Tw. containing also a little acetic acid. Then wash well in fresh water and pass through the dilute soap bath as described above (62). It will be noticed that some chlorine is given off in the bleach bath containing the acetic acid, but the bleaching does not require the after use of an acid, from which there is always danger of tendering the fibre.

**Experiment 27. Use of Lime Boil in Bleaching Cotton.** — Prepare a solution of lime-water by slaking 10 grams of quick-lime (oxide of calcium, CaO) with a small quantity of water, and then diluting to 300 cc. Boil a weighed skein of cotton yarn in this bath for 1 hour, then wash and pass through a cold bath containing 300 cc. of water and 3 cc. of concentrated hydrochloric acid; work for 15 minutes. Wash and bleach for 1 hour in a cold solution of chloride of lime at 2° Tw. Wash, and pass back through the cold acid bath for 15 minutes. Then wash well and soap as usual in a dilute warm soap bath. Wash and dry (63).

Reweigh and calculate the percentage of loss. Compare this method of bleaching with the previous ones. The first treatment with acid is required in order to dissolve out any lime compounds formed in the fibre, which would otherwise remain and tender the cotton, and also not allow the chloride of lime to act as perfectly as it should.

**Experiment 28. Use of Sodium Hypochlorite.** — Prepare a bath containing sodium hypochlorite solution of 1° Tw. In this cold bath steep for 1 hour a skein of cotton yarn previously boiled out with caustic soda. Wash and pass through a cold bath of sulphuric acid at 1° Tw. for 15 minutes. Then wash well and soap as usual (64). Sodium hypochlorite is prepared by adding a solution of soda ash to one of chloride of lime, allowing the precipitate of calcium carbonate to settle and drawing off the clear liquor. It is more efficient as a bleaching agent than chloride of lime, and is also less liable to cause tendering of the fibre. It is more expensive, however, than chloride of lime.

**Experiment 29. Comparison of the Use of Sulphuric and Hydrochloric Acids in Bleaching Cotton.** — Take two skeins of cotton yarn which have been boiled out with caustic soda and bleach them in the usual manner with chloride of lime solution at 2° Tw. Without washing, take one of the skeins and pass through a bath containing 300 cc. of water and 3 cc. of hydrochloric acid cold for 15 minutes, then wash well and dry (65). Take the other skeins and treat in a similar manner with a solution of 2 cc. of sulphuric acid in 300 cc. of water; wash well and dry (66). Notice that in the bath containing the hydrochloric acid there is no precipitate formed, as the lime compound with this acid is soluble in water; whereas in the sulphuric acid bath a precipitate of calcium sulphate is formed which will remain to a greater or lesser extent in the cotton.

**Experiment 30. Use of Chlorozone.** — Prepare a bath containing 300 cc. of water and 20 cc. of "chlorozone," and bleach a skein of cotton yarn which has been boiled out in caustic soda in this bath for 1 hour. Enter the skein at a temperature of about 200° F. and allow to cool in the bath without further heating.



Then squeeze, and pass through a bath of sulphuric acid solution at 1° Tw. cold for 15 minutes. Wash well and dry (67).

#### NOTES.

**1. General Method of Cotton Bleaching.** — Ordinary American cotton is of a comparatively white color when in the natural raw state; but, nevertheless, it contains a small amount of natural pigment matter of a yellowish brown color. This pigment is so small in amount that it does not interfere in the general dyeing of cotton; but when light, delicate shades are desired in dyeing, or when the cotton material is to be left in the white condition for sale, it is usually necessary to bleach it. Cotton in the loose state is very seldom bleached, since the bleaching processes considerably deteriorate the spinning qualities of the fibre by removing its waxy coating; the fibre is also made more brittle by the bleaching which causes a largely increased amount of waste in carding and spinning; furthermore, after bleached cotton is passed through the numerous mechanical operations of carding and spinning it will become more or less discolored and will have acquired considerable dirt, so that the final yarn or cloth would be unsatisfactory as a bleached product. Yarn is sometimes spun from bleached stock for the manufacture of knit goods, thus giving a half-bleached product; it is also used for half-bleached filling yarns. Cotton yarn is frequently bleached both for the purpose of being dyed in delicate shades and of being manufactured into white goods — more especially knitted fabrics, lace, etc. The chief form, however, in which cotton is bleached is that of cloth; in which case it may be used (a) as a *bleached bottom* for the dyeing of delicate shades or for colors such as Turkey-red, (b) for *print cloth* in the many processes of calico-printing, and (c) for the purpose of being sold in the white state, or as a *market-bleach*.

Though a number of chemical agents have been suggested for the bleaching of cotton, the one which has been most successfully and extensively employed is *chloride of lime* or *bleaching powder*. The effective bleaching agent in the chloride of lime is chlorine



in a loosely combined condition. But the chlorine itself does not accomplish the bleaching in a direct manner. In the process the chlorine is liberated in the nascent condition in the presence of water; the latter is decomposed by the chlorine yielding hydrochloric acid and nascent oxygen, and it is this oxygen which causes the bleaching action. The chemical reactions may be thus represented:

Chloride of lime  $\rightarrow$  chlorine.

Chlorine + water  $\rightarrow$  hydrochloric acid + oxygen.

Chlorine of itself is without any bleaching action, a fact which has been demonstrated by allowing *dry* chlorine to act on sensitive colors, the result being that the colors were not destroyed.

**2. The Operations in Cotton Bleaching.** — There are five distinct operations in the proper bleaching of cotton:

(1) *Boiling-out*; this is really a scouring operation, the object of which is to remove all the waxy and resinous matters in the fibre.

(2) *Treatment with bleaching powder solution*; this is for the purpose of destroying the natural coloring-matter in the fibre, and also for the breaking down of various non-cellulosic matters associated with the cellulose of the cotton.

(3) *Treatment with a dilute solution of acid*; this is generally termed "souring," and is for the purpose of dissolving the lime compounds in the fibre left from the bleaching powder and to decompose any chlorine compounds which may have been formed.

(4) *Washing*; this is for the purpose of removing all soluble matters resulting from the action of the bleaching powder and the acid; also for the removal of the acid from the fibre.

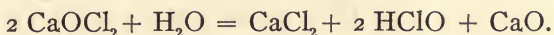
(5) *Soaping and tinting*; this is for the purpose of neutralizing the last traces of acid, and also for softening the cotton. The tinting is to give a slight bluish tone to the white.

**3. Boiling-out.** — The scouring of cotton intended for bleaching must be carried out much more thoroughly than when the operation is merely for the purpose of wetting-out the cotton

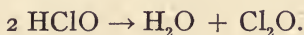
previous to dyeing. In the latter case it is only necessary that the external waxy coating on the fibre be removed or softened in order that water may easily impregnate the cotton. But in boiling-out for bleaching it is required to remove very completely all the impurities in the fibre, including the waxy coating, the miscellaneous resinous matters, the albuminous substances, and in fact all matters of a non-cellulosic character. It is the object in bleaching to obtain a practically pure cellulose for the bleached cotton. For the wetting-out of cotton, a dilute solution of soap, soda ash, or soluble oil only is required, but for the proper boiling-out of the cotton a rather strong solution of caustic alkali or soda ash is required; the time of boiling is much prolonged (usually 7 to 10 hours), and it is generally conducted under pressure in a closed kier. This very thorough boiling-out of the cotton previous to bleaching is necessitated by the fact that if any resinous matters (or so-called *pectin*) are left in the fibre, the bleached material will gradually become yellow on exposure to light and air. Formerly *lime* was very generally used for the boiling-out of cotton, in which case it was necessary to pass the material through an acid bath (so-called *gray sour*) to remove particles of lime which might otherwise "burn" the fibre. It was thought that boiling with lime caused a more perfect decomposition and removal of the resinous substances in the cotton. Lime, however, is not so much used at the present time, it being replaced by caustic soda, the action of which is more efficient and requires less time. When the lime boil is used (chiefly with piece-goods) a previous boiling with *resin soap* is usually given. *Silicate of soda* is a very good alkali for boiling-out cotton for bleaching. Many popular "bleach assistants" consist of varying proportions of silicate of soda, soda ash, and caustic soda. Silicate of soda does not give the cotton such a harsh feel as when caustic soda is used.

**4. Bleaching Powder and its Use.** — Bleaching powder or chloride of lime is prepared by treating slaked lime with chlorine gas. Its chemical formula is  $\text{Ca} \begin{smallmatrix} \text{OCl} \\ \diagdown \\ \text{Cl} \end{smallmatrix}$ , or  $\text{CaOCl}_2$ . It is

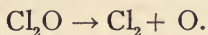
commonly known as "chemic" or "bleach." Chloride of lime is a yellowish white powder which smells strongly of chlorine, especially if moistened. When treated with water it partly goes into solution and partly forms a bulky white precipitate consisting for the most part of lime (CaO). The solution has a yellowish color and is the liquid employed for the preparation of the bleaching bath. A good quality of chloride of lime should contain about 36 per cent. of available chlorine, that is to say, chlorine which is active in the bleaching process. The exact chemical reactions which take place in the use of chloride of lime are not thoroughly understood, though they have been the subject of much investigation. It is probable that when chloride of lime is dissolved in water, calcium chloride (CaCl<sub>2</sub>), hypochlorous acid (HClO), and calcium oxide (CaO) are formed, as follows:



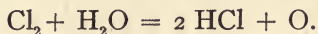
Hence the bleaching liquor, as used, may be considered as a solution of hypochlorous acid; the calcium chloride produces no effect in bleaching. No doubt a portion of the lime also remains in solution as calcium hypochlorite (Ca(OCl)<sub>2</sub>). The insoluble calcium oxide is filtered off (or settled out) before the bleaching liquor is used. Hypochlorous acid is a very unstable substance (especially in the presence of organic matter, such as the fibres), and it readily decomposes into water and an oxide of chlorine (Cl<sub>2</sub>O), as follows:



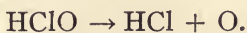
The latter is a strong oxidant, as it splits up into chlorine and free oxygen:



The chlorine thus liberated reacts with the water present to form hydrochloric acid and another portion of free oxygen:



Under certain conditions it is probable that the hypochlorous acid decomposes directly into hydrochloric acid and oxygen:





Solutions of bleaching powder are best prepared by first grinding the powder with a small quantity of cold water until a thin uniform paste is obtained, and then diluting with cold water and allowing to settle until the liquor is clear. A concentrated solution of bleaching powder will show a density of about 18° Tw. For the preparation of the bleaching bath this is diluted to about 2° Tw. Care should be taken that no undissolved particles of bleaching powder pass into the bleaching bath, otherwise the cotton may become tendered in spots. It is also necessary that the material be completely immersed in the solution during the bleaching, for under the influence of the oxygen of the air the bleaching liquor will seriously weaken the cotton. The temperature of the bleaching bath should always be cold; it is only in exceptional cases where low grade material is treated that the bleaching liquor is ever warmed, and even then only to about 100° F. The time of immersion of the cotton in the bleaching solution should be from one-half to one hour; too long a treatment will cause a tendering of the fibre.

**5. The Acid Treatment.** — When the cotton comes from the solution of bleaching powder it contains a considerable amount of lime compounds, partly as calcium hypochlorite and partly as calcium oxide; there is also present calcium chloride. The acid treatment (generally known as "souring") is for the purpose of decomposing the calcium hypochlorite and the calcium oxide:



In the first case hypochlorous acid is formed which furthers and completes the bleaching. In both cases calcium sulphate (gypsum) is formed as a white, finely divided, though insoluble, powder. This is quite easily removed from the fibre by subsequent washing, and being of a very neutral character, has no action on the cotton. As a rule, the cotton comes up much whiter after the souring, and the evolution of free chlorine gas is very evident. The souring is usually done in a cold bath of sulphuric acid of 1° Tw. density. Stronger solutions are not



advisable, as they are liable to weaken the cotton. Hydrochloric acid may be used to replace the sulphuric, in which case calcium chloride will be formed, which is a very soluble salt and is more easily removed from the fibre than the insoluble calcium sulphate. To obtain an equivalent acid strength about 2.25 parts by weight of hydrochloric acid should be used for 1 part by weight of sulphuric acid. In case the boiling-out, bleaching, etc., are carried out in machines containing copper or bronze a small amount of copper salt will be formed which with sulphuric acid will produce an insoluble precipitate of a double sulphate of copper and calcium. This will become fixed in the cotton and is very difficult to remove. If hydrochloric acid, however, is used, no insoluble precipitate will be formed, and the copper salt is easily washed away.

**6. Washing.** — Immediately following the souring the cotton should be thoroughly washed with fresh water in order to remove as far as possible all of the acid. Should the washing be delayed for any length of time there is danger of portions of the bleached material becoming dry, which will cause tender spots to form. The washing should be continued until the presence of acid is no longer evident; this may be shown by testing the cotton with a piece of blue litmus paper, which will turn red if any acid is present. The washing is also for the purpose of removing the sulphate of calcium which is precipitated in the cotton during the souring. The chlorine which is generated in the material during the same process is also removed by the washing, and care should be taken to eliminate it very thoroughly, otherwise the cotton will subsequently be weakened by over-oxidation and the formation of acid in the fibre. The presence of chlorine in the cotton may be tested for by a mixed solution of potassium iodide and starch paste, which will give a blue color with a trace of chloride. This test depends on the fact that chlorine liberates iodine from potassium iodide, and the free iodine combines with the starch to form a compound with an intensely blue color.

**7. Soaping and Tinting.** — The final operation essential to the bleaching of cotton is that of soaping. For this purpose the

material is treated in a dilute lukewarm solution of soap. The latter should be of good quality and free from any ingredients liable to cause discolorations in the dried and finished bleach. The object of the soaping is primarily to *soften* the cotton, which will have acquired considerable harshness in the boiling-out, bleaching, and acid treatments. It also has the purpose of neutralizing absolutely all trace of acid in the cotton, and thus preventing subsequent tendering. In the soap bath it is also customary to add a small quantity of a blue dyestuff, such as Cotton Blue, Methylene Blue, Soluble Prussian Blue (bleacher's tint), etc., for the purpose of *tinting* the bleached white to a satisfactory bluish tone. In case a cream-white bleach is desired, the tinting is omitted. Care must be had not to tint the cotton too strongly, otherwise the material will appear dull and dirty.

In the entire process of bleaching, the cotton will lose in weight about 5 to 7 per cent. If properly bleached the loss in tensile strength should not be over 5 per cent. The elasticity will be somewhat less than that of the unbleached cotton. The tendering of cotton in bleaching may be due to several causes:

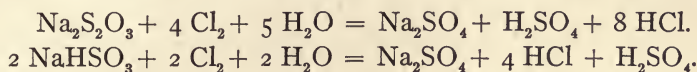
(1) Oxidation caused by exposure to the air during the boiling-out process. If a skein of cotton yarn is so hung as to be partly suspended in a solution of caustic soda and boiled thus for some time, it will be found to be seriously weakened at that part where it comes into contact simultaneously with the alkaline liquor and the air.

(2) Oxidation due to the use of too strong a solution of bleaching powder, or to its becoming overheated.

(3) The drying of acid in the fibre or of particles of lime from sediment in the bleaching bath.

The oxidation of cotton leads to the formation of a substance known as *oxycellulose*, which is structureless and pliable in character, hence its formation leads to a weakening of the fibre. The presence of oxycellulose may usually be recognized by staining the cotton with a dilute solution of Methylene Blue; ordinary cotton has but slight affinity for this coloring-matter, whereas oxycellulose is strongly dyed.

8. Use of "Anti-chlor." — As the perfect removal of the chlorine from the cotton is very difficult by simply washing with water, it is sometimes expedient to neutralize the free chlorine with a suitable chemical agent. The chief substances used as "anti-chlors" are sodium hyposulphite ( $\text{Na}_2\text{S}_2\text{O}_3$ ) and sodium bisulphite ( $\text{NaHSO}_3$ ). The reactions in the two cases are as follows:



After treatment with anti-chlor the bleached cotton should be well washed and soaped, for it will be noticed from the above reactions that acid is formed in both cases.

9. Bleaching Loose Cotton. — Loose cotton is seldom bleached for purposes of spinning, as the bleaching operation considerably deteriorates the spinning qualities of the fibre. This is due to the fact that in the bleaching the waxy matters are removed, and hence the fibre becomes less plastic and coherent, besides being more brittle. Sometimes, however, cotton in the half-spun condition is bleached, and there are mechanical devices available for the proper bleaching of cotton roving and slubbing. A cold method for bleaching loose cotton has been proposed wherein the cotton is first treated in a suitable machine so that cold water is forced through the mass under considerable pressure; then a cold solution of bleaching powder is circulated through the cotton, and subsequently dilute acid, followed by a thorough washing and soaping. This method is said to leave the cotton almost unimpaired as to its spinning qualities. Such a process is attaining considerable practical value for the spinning of filling cops from bleached stock.

Loose cotton, however, is largely bleached for use as medicinal *absorbent cotton*. The object in view in this case is not only to obtain a white and pure fibre, but also to make it highly absorbent of liquids. In fact, the purpose is not so much to bleach the cotton in the sense of destroying the color, as to remove all impurities from the fibre which may in any manner interfere with its ready absorption of liquids. Hence, the chief operation is a



very thorough boiling-out to remove perfectly the waxy and resinous matters. For this purpose the cotton is boiled in a comparatively strong solution of caustic soda under pressure for 8 to 10 hours. After this treatment it is bleached in the usual manner with chloride of lime and sulphuric acid. The quality of absorbent cotton is tested by the readiness with which it sinks in water.

**10. Bleaching with Acetic Acid.** — In some cases cotton is bleached with the use of acetic rather than sulphuric acid. Acetic acid is less liable to cause tendering of the fibre, and the acid may be added directly to the bath of chloride of lime, though this causes a considerable loss of chlorine. Otherwise the acetic acid acts in the same manner as sulphuric acid. Although this method has been strongly advocated by some chemists it does not seem to have acquired much practical importance.

**11. Bleaching with Sodium Hypochlorite.** — This reagent is also known as "chloride of soda," and corresponds to chloride of lime in its bleaching properties. It may conveniently be prepared by adding a solution of soda ash to one of chloride of lime until no further precipitation takes place. The white sediment of calcium carbonate is allowed to settle and the clear liquor containing sodium hypochlorite in solution is drawn off and used for bleaching. It is sometimes used for the bleaching of fine and delicate fabrics and where it is not desirable to introduce any lime into the cotton.

A bleaching agent similar to sodium hypochlorite is prepared by saturating a cold solution of caustic soda with chlorine gas. This solution is prepared under a variety of names such as *chlorozone*, *oxychlorine*, etc. It usually has a pink color, due to the presence of certain impurities in the caustic soda forming colored salts with the chlorine. It is used in the same manner as sodium hypochlorite and is a very efficient bleaching agent. Solutions of sodium hypochlorite prepared electrolytically by the action of the electric current on a solution of common salt are also employed in bleaching. It is claimed that liquors thus prepared show a much higher bleaching efficiency than ordinary solutions of sodium hypochlorite.



## SAMPLES.

52. Cotton yarn boiled-out with caustic soda and bleached.
53. Cotton yarn boiled-out with soap and bleached.
54. Cotton yarn boiled-out with Monopol Soap and bleached.
55. Bleached yarn treated with anti-chlor.
56. Loose cotton in raw state.
57. Loose cotton bleached and absorbent.
58. Bleached yarn untinted.
59. Tinted with  $\frac{1}{100}$  per cent. of dyestuff.
60. Tinted with  $\frac{1}{100}$  per cent. of dyestuff.
61. Tinted with  $\frac{1}{50}$  per cent. of dyestuff.
62. Cotton yarn bleached with use of acetic acid.
63. Cotton yarn boiled-out with lime and bleached.
64. Cotton yarn bleached with sodium hypochlorite.
65. Bleaching with use of hydrochloric acid.
66. Bleaching with use of sulphuric acid.
67. Cotton yarn bleached with chlorozone.

## RECORD OF RESULTS.

No. Test.	Weight before Bleaching.	Weight after Bleaching.	Loss in Weight.	Percentage of Loss.
22(a).....				
22(b).....				
22(c).....				
24.....				
27.....				

## QUIZ 4.

82. What is the natural color of raw cotton? For what purposes is it necessary to bleach cotton? In which form, loose stock, yarn, or cloth, is cotton mostly bleached?

83. What bleaching agent is principally used in connection with cotton? Explain the principle of its bleaching action.

84. What are the several operations necessary in the bleaching of cotton? Explain the function of each process.

85. Why must the boiling-out of cotton before bleaching be very thorough? What materials are removed in the boiling-out process?

86. What is chloride of lime? Under what other names is it known in bleaching?

87. Explain the various chemical reactions taking place when bleaching with chloride of lime.

88. What reaction occurs when chloride of lime is dissolved in water? How are bleaching powder solutions prepared?

89. At what density is the solution of chloride of lime usually employed for bleaching purposes? Why should the cotton be completely immersed in the liquor?

90. For what length of time is the cotton steeped in the bleaching powder solution and at what temperature?

91. Is the decolorization of the cotton complete after steeping in the chemical solution? What after-treatment is necessary?

92. What is meant by a "sour"? How is the souring done? Explain the reaction of sulphuric acid on chloride of lime.

93. The acid solution employed for souring is used at what strength? The odor of what gas is noticed when the cotton is soured?

94. Why is it necessary to wash the cotton very thoroughly after souring? How would you test for the presence of acid in the bleached cotton? For the presence of chlorine?

95. How is the test solution for detecting chlorine prepared? Explain the reaction which takes place in this test.

96. How much does cotton yarn lose in weight by bleaching? If properly bleached does cotton yarn lose much in tensile strength?

97. Give the different causes which may lead to the tendering of cotton in the operations of bleaching. What is oxycellulose, and how may its presence in bleached cotton be detected?

98. What is meant by an "anti-chlor," and for what purpose is it employed in bleaching? What anti-chlors are chiefly used? Explain the chemistry of their action.

99. For what purpose is loose cotton chiefly bleached? How is the bleaching carried out? What is the difference in the behavior towards cold water of loose raw cotton and loose bleached cotton?

100. How is bleached cotton tinted? About how much dyestuff is required for proper tinting?

101. What is the purpose of "softening" bleached cotton, and how is this done?

102. In what manner may cotton be bleached with the use of acetic acid? Explain the chemistry of this process.

103. How is cotton boiled-out with lime? What treatment is necessary after the boiling-out, and why? Why has the lime boil been recommended?

104. What is sodium hypochlorite? How is it prepared? How is it employed for the bleaching of cotton?

105. How does sodium hypochlorite compare with chloride of lime as a bleaching agent?

106. What difference is to be noticed in the use of sulphuric and hydrochloric acids in the bleaching of cotton? Give the chemical reactions which take place in both cases.

107. What are chlorozone and oxychlorine? How are these prepared? How are they employed in bleaching?

## SECTION V.

### CLASSIFICATION OF DYES.

**Experiment 31. Action of Acid Dyes.** — Prepare a bath containing 300 cc. of water and 5 cc. of Formyl Violet solution. Take a scoured test-skein of woolen yarn, wet it out with warm water, and place it in the above bath; boil for one-half hour; then wash well in fresh water and dry (68). Repeat this test, using a bath containing 300 cc. of water, 5 cc. of Formyl Violet solution, and 10 cc. of sulphuric acid solution. It will be found that in the first test the wool is only slightly dyed, whereas in the second test it is well dyed (69). Repeat the second test, using a wet-out test-skein of cotton yarn; wash well and dry (70). It will be found that the cotton is only slightly tinted by the acid dyestuff. Repeat the test again, using a skein of boiled-off silk, and it will be found that the silk (71) is dyed like the wool.

**Experiment 32. Action of Basic Dyes.** — Prepare a bath containing 300 cc. of water and 10 cc. of Magenta solution, and boil a test-skein of woolen yarn therein for one-half hour, then wash well and dry (72). It will be noticed that the dyestuff is taken up by the wool directly. Repeat the test, using a skein of silk. It will be found that the silk (73) also dyes directly with the basic coloring-matter. Repeat the test again, using a skein of cotton yarn. It will be found that in this case the cotton (74) is only slightly tinted with the dyestuff. Take a second skein of cotton and work it for one-half hour in a bath containing 300 cc. of water and 10 cc. of tannic acid solution at 180° F. Squeeze, and then dye as before described in a fresh bath. It will now be found that the treated cotton (75) will combine with the basic dyestuff.

**Experiment 33. Action of Substantive Dyes.** — Prepare a dye-bath containing 300 cc. of water and 10 cc. of a solution of



Benzopurpurin 4B, and boil a test-skein of wool therein for one-half hour, then wash well and dry (76). It will be noticed that the wool combines directly with the substantive dyestuff.

Repeat the test, using a skein of silk, and it will be found that the silk will also be dyed (77). Repeat the test again, using a skein of cotton yarn; the cotton will also be dyed (78).

**Experiment 34. Action of Mordant Dyes.** — Dye a skein of woolen yarn in a bath containing 300 cc. of water and 10 cc. of Alizarin Red; boil for one-half hour. It is found that the dyestuff is not taken up to any extent by the fibre (79). Boil a second skein of woolen yarn in a bath containing 10 cc. of chrome solution (potassium bichromate) for one-half hour; then rinse and dye as above given (80). The dyestuff will now be absorbed by the mordanted wool, combining with the chromium oxide in the fibre to form a color-lake. Dye a skein of cotton yarn with Alizarin Red in the above manner; it will be found that the fibre has no attraction for the dyestuff (81). Mordant a second skein of cotton yarn by working in a cold solution containing 200 cc. of water and 5 grams of ferric chloride for 10 minutes; squeeze and pass through a cold solution containing 200 cc. of water and 2 grams of soda ash. This furnishes a deposit of iron oxide on the fibre and gives the latter a buff color (82). Now dye this mordanted skein in the above manner with Alizarin Red, and it will be found that the dyestuff is absorbed and the cotton becomes dyed (83). Dye a skein of silk with the solution of Alizarin Red; it will be noticed that silk is similar to wool and cotton, and is not dyed (84) by the alizarin dye. Mordant a second skein of silk with chrome in the same manner as was used for wool, and then dye with the Alizarin Red. It will be found that the mordanted silk (85), like the wool, will combine with the dyestuff.

**Experiment 35. Action of Pigment Dyes.** — Work a skein of cotton yarn for 15 minutes in a cold solution containing 200 cc. of water and 5 grams of lead acetate. Squeeze and then work for 15 minutes in a second cold solution containing 200 cc. of water

and 2 grams of potassium bichromate. Wash well and dry (86). A yellow pigment consisting of chrome yellow (chromate of lead) will be formed in the fibre through the chemical reaction between the lead acetate and the potassium bichromate. This pigment is purely of mineral nature and is simply deposited in the cells of the fibres and does not combine with the substance of the fibre itself, as is the case with the other methods of dyeing.

#### NOTES.

1. **General Classification of Dyestuffs.** — With respect to their general properties nearly all coloring-matters may be divided into four general classes, as follows:

- |                 |                       |
|-----------------|-----------------------|
| (a) Acid Dyes.  | (c) Substantive Dyes. |
| (b) Basic Dyes. | (d) Mordant Dyes.     |

This classification in a general way is based on the chemical nature of the dyestuff and its reaction towards the fibre. The following is a brief summary of these properties:

(a) *Acid Dyes.* Salts of color-acids; dye animal fibres directly; do not dye vegetable fibres; mostly applied to wool and silk.

(b) *Basic Dyes.* Salts of color-bases; dye animal fibres directly; dye vegetable fibres on a tannin mordant; mostly applied to cotton and silk.

(c) *Substantive Dyes.* Of neutral chemical nature; dye both animal and vegetable fibres directly; mostly applied to cotton and somewhat to both wool and silk.

(d) *Mordant Dyes.* Of neutral chemical nature; dye neither animal nor vegetable fibres directly, but require a metallic mordant; mostly applied to wool.

The great majority of the dyestuffs used at the present time are derived from coal tar, the vegetable dyes, with few exceptions, being almost obsolete.

- |   |
|---|
| (a) <i>Acid Dyes.</i> Mostly derived from azo compounds of benzene. |
| (b) <i>Basic Dyes.</i> Mostly derived from aniline.                 |
| (c) <i>Substantive Dyes.</i> Mostly derived from benzidine.         |
| (d) <i>Mordant Dyes.</i> Mostly derived from anthracene.            |

Previous to the discovery of the coal-tar dyes, the coloring-matters employed were of either vegetable or mineral origin.

(a) *Vegetable Dyes.* Logwood, fustic, hypernic, cochineal, madder, cutch, camwood, etc.

(b) *Mineral Dyes.* Iron black, iron buff, manganese bistre, chrome yellow, etc.

The first coal-tar dyestuff was discovered in 1856 by Perkin; it was known as Mauve, and was soon followed by other aniline dyes. It is wrong, however, to apply the term "aniline" colors to all coal-tar dyes, as there are now many which are not derived from aniline.

For the same amount of coloring-matter the coal-tar dyes are much cheaper than the vegetable colors; they have also far greater intensity of color, are much brighter, and in general are faster.

Based on their general methods of application, we may classify practically all of the dyes used at the present time into the following groups:

(a) *Acid dyes*, such as Acid Violet, Naphthol Yellow, etc.

(b) *Basic dyes*, such as Magenta, Methylene Blue, etc.

(c) *Mordant dyes*, such as Alizarin Red, Gallocyanine, etc.

(d) *After-chromed dyes*, such as Chromotrop, Diamond Black, etc.

(e) *Substantive dyes*, such as Benzopurpurin, Chrysophenine, etc.

(f) *Developed dyes*, such as Diamine Black BH, Primuline, etc.

(g) *Naphthol dyes*, such as Paranitriline Red, etc.

(h) *Coupled dyes*, such as Benzo Nitrol Brown, etc.

(i) *Sulphur dyes*, such as Immedial Black, Katigen Green, etc.

(j) *Vat dyes*, such as Indigo, Indanthrene Blue, etc.

(k) *Oxidized dyes*, such as Aniline Black.

(l) *Mineral pigment dyes*, such as Chrome Yellow, Prussian Blue, etc.

The most important of these classes, and those containing the greatest number and the most diversified colors, are the four groups mentioned in the first paragraph of this section.



The *acid dyes* are principally used for the dyeing of wool and silk, and only to a limited extent for the dyeing of cotton or other vegetable fibres. They are applied to the animal fibres in baths containing either sulphuric acid or acetic acid. The *basic dyes* are used chiefly for the dyeing of cotton and silk; only a few members of this group are used in wool-dyeing. They are applied to the animal fibres directly from neutral baths. For cotton, or other vegetable fibres, a mordant of an acid character (such as tannic acid) is required. The *mordant dyes* are almost exclusively used for the dyeing of wool, with the exception of Alizarin Red, which is also largely used for the dyeing of cotton (for Turkey-red). These dyes do not have a direct affinity for any of the fibres, and require the use of a metallic mordant (usually potassium bichromate) in their application. The *after-chromed dyes* are very similar to the mordant dyes in their general characteristics, but the mordant is applied after the dyeing. These colors are used exclusively on wool and have a direct affinity for this fibre, though the color so obtained, as a rule, has little value. The after-chroming process usually alters this color considerably and gives it fastness. The *substantive dyes* are so called because they have a direct affinity for all fibres. They are primarily cotton dyes, though they are also used to some extent on wool and silk. They are applied in neutral baths. The *developed dyes* are used almost exclusively on cotton, though a few are also applicable to silk. They are first dyed in a manner similar to the substantive colors in a neutral solution; the dyed material is then treated with a solution of nitrous acid (obtained by the addition of acid to a solution of sodium nitrite), — a process known as *diazotizing*,—and afterwards with a solution of beta-naphthol (or other similar body), which is known as the *developer*, and from which this class of dyes receives its name. By these operations a new dyestuff is built up in the fibre. The *naphthol dyes* are somewhat similar in general to the preceding group, only the order of the operations is reversed. The material to be dyed is first treated with a solution of beta-naphthol (or other similar developer), and then with a solution of a diazotized



base representing the dyestuff. In this manner a dyestuff is actually made in the fibre. These dyes are but few in number and are applied only to cotton. The *coupled dyes* are another similar group, the material being first dyed with a substantive color and then treated with a solution of diazotized paranitraniline; this resulting in the formation of a new dyestuff in the fibre. Such dyes are applicable only to cotton and are quite limited in number and range of color. The *sulphur dyes* are also applied almost exclusively to cotton, though in certain cases they may also be used on silk. They are dyed with the aid of sodium sulphide which is added to the dye-bath, and the dyestuff apparently contains sulphur compounds in its composition. In other respects they are very similar to the substantive dyes. The *vat dyes* form a small group of colors employed on all the fibres. The dyestuffs themselves are insoluble and require to be first reduced by means of a strong reducing agent (such as sodium hydrosulphite or other suitable substance) and dissolved in an alkaline liquor. This combination forms the so-called "vat." Indigo is the chief representative of this group, though other dyes of a similar character have lately been added, such as the indanthrene, dianthrene, and algol colors, as well as the thio-indigo colors. The group of *oxidized dyes* is practically limited to only one member known as Aniline Black. This dye, which is extensively used, is formed by the proper oxidation of aniline directly in the fibre. It is used principally on cotton and to a lesser extent on silk. The *mineral pigment dyes* are colored compounds of the metals formed by the precipitation in the fibre of suitable metallic salts, such as chrome yellow formed by precipitation of lead acetate in the fibre with potassium bichromate. These dyes are almost entirely used for dyeing cotton; some were formerly used for dyeing wool and silk, but this application of them is now almost entirely discontinued.

2. **General Theory of Dyeing.** — There have been two main theories to explain the general process of dyeing. The *chemical theory* supposes that dyeing involves a chemical reaction between the fibre and the dyestuff and that a definite chemical compound

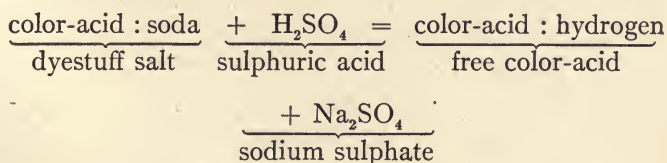
known as the *color-lake* is thus produced. The *mechanical theory*, on the other hand, considers the effect of dyeing to be simply a deposit of colored particles in the substance of the fibre, and the combination so formed to be merely a mechanical mixture. The chemical theory is supported by the facts that the animal fibres (wool and silk) exhibit well-defined chemical reactivities of an acid and a basic character, and that these fibres readily dye with the acid and basic dyestuffs; whereas cotton, which is practically inert as far as chemical reactivity is concerned, shows no pronounced attraction for these dyes; but if an acid mordant is added to the cotton fibre then the latter exhibits the power of combining with basic dyes, or if a basic mordant (a metallic oxide) is added the cotton will be able to combine with the acid dyes. Furthermore, in the case of mordant dyes, the combination between the dyestuff and the mordant may be made independent of the fibre, and a chemical reaction undoubtedly takes place in the formation of such a color-lake. On the other hand, it may be shown that the substantive colors dye cotton quite readily without any evidence of a chemical reaction. Also almost any porous substance (even such substances as unglazed porcelain, finely divided silica, etc.) will take up a dyestuff from solution (especially the basic dyes) and become truly dyed thereby. Even unmordanted cotton will dye with many of the acid and basic dyes if concentrated solutions are employed, and the colors so obtained have a certain degree of fastness. With the present knowledge of dyeing, it seems more reasonable to assume that the substance of the fibres is capable of *dissolving* such bodies as dyestuffs and mordants, bringing about a condition which we know as *solid solution*, which merely means that one solid substance is dissolved in another solid. According to this view of regarding the phenomena of dyeing, the dissolved coloring-matter in its water-solution passes into a fibre-solution. There are many factors influencing the degree and rapidity of this form of solution, among which the most important appear to be the chemical activity existing between the dyestuff (or mordant) and the fibre, the heating of the dye-bath, the presence of various

chemicals in the dye-bath or fibre, and the mass-relations between the fibre, the dye-bath, and the dyestuff.

Though we cannot regard the chemical activity of the fibre toward the dyestuff as primarily the cause of dyeing, nevertheless there can be no doubt but that this factor often exerts a determining influence in the process. This is especially true when we consider the chemical relations between the acid and basic dyes and the animal fibres wool and silk. The chemical combination possible between the fibre and the dyestuff in this case no doubt determines the fixation of the solution of the coloring-matter in the substance of the material dyed.

Heat also appears to play an important rôle in dyeing as it does in all forms of solution. By elevating the temperature the mobility of the molecular aggregates of both the dyestuff and the fibre is increased so as to allow of a more intimate mixture of these molecules with one another. The rapidity and degree of dyeing is nearly always greater in a hot dye-bath than in a cold one; and the proper regulation of this temperature permits the dyer to so regulate the taking-up of the color by the fibre as to obtain even and well-penetrated dyeings.

The presence of certain chemicals in the dye-bath also has an important influence in the regulation of the dyeing. In the case of acid dyes, the presence of acid (such as sulphuric or acetic) liberates the free color-acid from the dyestuff salt and thus promotes and accelerates the dyeing by allowing of the ready and complete combination between the fibre-base and this color-acid. We may represent this reaction somewhat in the following graphical manner:



Color-acid + wool-base = color-lake.



The basic dyes appear to become dissociated when dissolved in water; that is to say, the color-base of these dyes becomes spontaneously separated from the acid with which it is combined in the form of its dyestuff-salt; and the free color-base thus formed combines readily with the acid component of the fibre to form the color-lake. Therefore the basic dyes can be applied to the animal fibres in a neutral dye-bath. As a rule, however, they are taken up too rapidly by the fibre to allow of even dyeing, so the bath is usually made slightly acid (with acetic acid) in order to retard the dyeing action.

The presence of glaubersalt (or other neutral salt such as common-salt) appears to influence the dyeing reaction by more perfectly distributing the dyestuff molecules through the fibre substance. This is more especially true of its action in reference to the acid and basic dyes. It may be said to impede the progress of the dyestuff molecules in their passage from the water solution to that of the fibre. The number of glaubersalt molecules is very large compared with the number of those of the dyestuff, and hence its action may be compared to that of a large crowd of people impeding the progress of a man in walking towards a definite point. The action of common-salt in the case of the substantive dyes appears to assume a somewhat different character; it reduces the solubility of the dyestuff in the liquid of the dye-bath and hence increases its relative solubility in the fibre, and thus helps to exhaust the bath by forcing more color on the cotton. The action of these chemicals appears to be about the same if they are contained in the fibre itself rather than in the dye-bath. If wool, for example, is treated with sulphuric acid, and washed, it appears to retain the sulphuric acid possibly in some weak form of combination. If such wool is placed in a bath containing an acid dyestuff, the dyeing reaction will proceed without further addition of acid.

The mass-relations existing between the fibre, the dye-bath, and the dyestuff also have an important influence in the dyeing reaction. It would be natural to expect that the greater the concentration of the dye-bath, that is, the greater the mass of the



dyestuff in proportion to the mass of the water, the more color will be taken up by the fibre. The same, of course, is true when the mass of the fibre is greater in proportion to that of the water.

**3. The Use of Mordants.** — It has been seen that certain dyes (such as the alizarin and anthracene series) do not form stable combinations with the fibres. If wool, for example, is boiled in a solution of Alizarin Red, in a certain sense it will become dyed, but the color may easily be washed from the fibre. In other words, though the dyestuff is soluble in the fibre, the color-lake does not become "fixed." These dyes, however, form very permanent color-lakes with many metallic oxides, such as those of aluminium, chromium, iron, etc. Furthermore, wool (and silk also) has the property of dissolving and fixing these metallic salts much in the same manner, for instance, as basic dyes are taken up by the fibre. Therefore if the animal fibres are first boiled in a solution of such metallic salt a certain quantity of the metallic oxide becomes dissolved and fixed in the substance of the wool (or silk), and the fibre so prepared can then be dyed a permanent color with the alizarin (or other mordant) dyestuffs. Cotton (and the vegetable fibres in general) does not have the property of dissolving and fixing these metallic salts (or mordants) to any extent; hence this method of mordanting and dyeing is not applicable to the vegetable fibres. The vegetable fibres do, however, possess the property of combining with tannic acid, and this furnishes a method of so preparing these fibres that they may be dyed with the basic dyestuffs. The salts of those metals which are more or less easily dissociated in boiling water are most applicable as mordants for the animal fibres. These salts include compounds of chromium, aluminium, iron, tin, copper, etc., the most important mordants for wool being potassium bichromate (chrome) and alum (or aluminium sulphate). Pyrolignite of iron (crude ferrous acetate) and the so-called nitrate of iron (really a ferric sulphate) are used largely for silk; although their use in this connection is primarily for purposes of weighting, their mordanting action being a secondary consideration. The color-lake in the case of a mordant dye consists of the triple compound:

fibre — metallic oxide — dyestuff. The mordant dyes are of a mild acid character or contain groups which permit of them uniting chemically with the basic metallic oxide. On account of the fact that cotton cannot be readily mordanted after the manner of wool, the general class of mordant dyes find little or no application to this fibre. About the only instance of their use in this connection is the dyeing of Turkey-red, and this requires a special and complicated process. One feature to be noticed in connection with the mordant dyes is that the same dyestuff often gives very different colors on different mordants; Alizarin Red, for instance, when dyed on a chrome mordant gives a rather dull purplish red; on an aluminium mordant it gives a bright red; on a tin mordant it gives a scarlet; and on an iron mordant it gives a dull purple.

**4. The Pigment Dyes.**—These coloring-matters are of a different nature from those of the other groups. While the latter are organic compounds and mostly derivatives from coal tar, the pigment dyes are of a mineral nature. They consist, really, of mineral pigments precipitated more or less mechanically in the fibre. Chrome yellow, for instance, consists of lead chromate, a compound of an intensely yellow color which may be prepared entirely independent of the fibre, and is used extensively as a pigment for the preparation of paints.

#### SAMPLES.

68. Wool dyed with acid dye without acid.
69. Wool dyed with acid dye with acid.
70. Cotton dyed with acid dye.
71. Silk dyed with acid dye.
72. Wool dyed with basic dye.
73. Silk dyed with basic dye.
74. Unmordanted cotton dyed with basic dye.
75. Mordanted cotton dyed with basic dye.
76. Wool dyed with substantive dye.
77. Silk dyed with substantive dye.
78. Cotton dyed with substantive dye.
79. Unmordanted wool dyed with mordant dye.
80. Mordanted wool dyed with mordant dye.

81. Unmordanted cotton dyed with mordant dye.
82. Cotton mordanted with an iron salt.
83. Mordanted cotton dyed with mordant dye.
84. Unmordanted silk dyed with mordant dye.
85. Mordanted silk dyed with mordant dye.
86. Cotton dyed with pigment dye.

### QUIZ 5.

108. Into what classes are dyestuffs in general divided? What is the difference between a dyestuff and a pigment?

109. From what substance are most of the dyes at present derived? What kinds of dyes were employed in former times?

110. When and by whom was the first coal-tar dyestuff made? Are all coal-tar dyes "aniline" dyes? How do the coal-tar dyes compare with the vegetable dyes in cheapness, intensity of color, brightness, and general fastness?

111. Give an outline of the chemical theory of dyeing. What facts argue for and against this theory?

112. What is the physical theory of dyeing? What is meant by a "solid solution"?

113. What is the chemical constitution of an acid dyestuff? Explain why acid dyes combine directly with the animal fibres and not with the vegetable fibres.

114. Why is acid added to the dye-bath in dyeing with acid dyes? Explain the chemical reaction which takes place between the acid and the dyestuff.

115. Which acid is mostly used in dyeing acid colors, and in what amount?

116. What is the eventual color-lake obtained in dyeing acid colors on wool or silk?

117. What is the chemical constitution of a basic dye? Explain the chemical difference between a basic and an acid dye.

118. Explain why the animal fibres combine directly with basic dyes while the vegetable fibres do not. How may the latter be so treated as to be capable of dyeing with the basic dyes?

119. Why is it not necessary to add either acid or alkali to the dye-bath in dyeing basic dyes? What is meant by "dissociation"?

120. What is meant by the term "substantive dye"? How does this class differ from the acid and basic dyes chemically?

121. How do substantive dyes react with wool, silk, and cotton? Is any addition to the bath necessary in dyeing these colors?

122. What is meant by a mordant? By a mordant dye? How do these dyes react with animal and vegetable fibres?

123. How may wool and silk be mordanted in order to dye with the mordant dyes? Can cotton be also mordanted in the same manner as wool? Why?

124. What class of metallic salts are employed as mordants? Briefly explain the chemistry of the mordanting process.

125. Of what does the color-lake consist when mordant dyes are employed? Why are mordant dyes not much applied to cotton?

126. Does Alizarin Red give the same color with a chrome as with an iron mordant? What is the color in each case?

127. What is meant by a pigment dye? How are these dyes obtained in the fibre?

128. Of what does Chrome yellow consist? How does it differ in chemical nature from the other dyes so far employed?

129. How is Chrome yellow applied to cotton? Give the chemical reactions which occur in its formation.

130. To what fibres are acid dyes mostly applied? Basic dyes? Mordant dyes? Pigment dyes? Substantive dyes?



## SECTION VI.

### APPLICATION OF ACID DYES.

**Experiment 36. General Method of Dyeing Acid Colors on Wool.** — Prepare a dye-bath containing 300 cc. of water, 20 per cent. of Glaubersalt, 4 per cent. of sulphuric acid, and 1 per cent. of Acid Magenta. Have the temperature of the bath at about 140° F. and place in it a well-scoured and wet-out test-skein of woolen yarn; by means of the stirring rods give the skein a few turns in the liquor so as to saturate the fibre thoroughly with the solution. Then allow the skein to hang from a stirring rod into the dye-bath, and heat the latter gradually to the boiling point, turning the skein from time to time so that it may dye up evenly. Do not maintain the liquor in a state of actual ebullition, as this will rapidly cause the fibres of the wool to felt together; keep the bath just at a simmer. Continue the dyeing at this temperature for one-half hour, turning the skein from time to time. Then remove the skein, squeeze out the dye liquor, rinse well in fresh water until no more color is removed from the fibre, then squeeze out and dry (87). This experiment represents the general method of dyeing nearly all acid dyes on wool.

**Experiment 37. Showing the Use of Glaubersalt in the Dye-bath.** — Prepare a bath containing 300 cc. of water, 4 per cent. of sulphuric acid, and 2 per cent. of Naphthyl Blue Black. Dye a test-skein of woolen yarn in this bath, entering at 120° F. and gradually raising to the boil, and continue at that temperature for one-half hour; then wash and dry (88). It will be found that the skein has become colored rather unevenly, due to the fact that no retarding agent such as Glaubersalt has been added. Now prepare a second bath similar to the preceding one, but also add 20 per cent. of Glaubersalt, and then dye a second skein

of woolen yarn as before (89). After washing and drying, compare the evenness of the colors on the two skeins.

**Experiment 38. Showing the Influence of the Amount of Acid in Dyeing with Acid Colors.** — Prepare four dye-baths, each containing 300 cc. of water, 20 per cent. of Glaubersalt, and 2 per cent. of Formyl Violet 10B. To the first bath add 1 per cent. of sulphuric acid, to the second add 2 per cent. of the acid, to the third add 4 per cent. of the acid, and finally to the fourth add 8 per cent. of the acid. Dye a skein of woolen yarn in each of these baths in the usual manner; that is, entering at 120° F., gradually raising to the boil and dyeing at that temperature for one-half hour. After dyeing, wash and dry, and compare the color on the several skeins (90, 91, 92, 93). Also compare the depth of color left in the respective dye-baths after the dyeing operation has been completed.

**Experiment 39. Showing the Exhaustion of the Dye-bath.** — Prepare a bath containing 300 cc. of water, 2 per cent. of Acid Magenta, 4 per cent. of sulphuric acid, and 20 per cent. of Glaubersalt. Dye a skein of woolen yarn in this bath in the usual manner. Squeeze the excess of dye liquor back into the bath; wash the dyed skein and dry (94). Then dye a second skein of woolen yarn in the same bath without any further addition of dyestuff or chemicals, but fill up the dye-bath with water so that the volume is brought back to the original point. After dyeing, squeeze the excess of liquor into the bath again, wash the second skein and dry (95). Then add water again to the bath to bring it back to the original volume, and dye a third skein of woolen yarn in the same manner, and after dyeing, wash and dry (96). Compare the color obtained on the three skeins, and this will give a good idea of the relative exhaustion of the dye-bath.

**Experiment 40. Dyeing Acid Dyes in a Neutral Bath.** — Some of the acid dyes are dissociated considerably on dissolving in water and liberate sufficient color acid to allow of the dyeing of the wool without the addition of any acid to the bath. Prepare a bath containing 300 cc. of water, 10 per cent. of Glaubersalt, and 1 per cent. of Orange ENZ. Dye a skein of woolen yarn in

this bath in the usual manner, and wash and dry (97). Prepare a second bath containing 300 cc. of water, 10 per cent. of Glaubersalt, 1 per cent. of Orange ENZ, and 4 per cent. of sulphuric acid. Dye a skein of woolen yarn in this bath in the usual manner, and wash and dry (98). Compare the color obtained on the two skeins by these methods.

**Experiment 41. Dyeing of Alkali Blue.** — The color acids of a few of the acid dyes are insoluble in water, and therefore acid cannot be added directly to the dye-bath, but must be employed in a separate bath. This method is represented in the application of Alkali Blue. Dye a test-skein of woolen yarn in a bath of 300 cc. of water, 10 per cent. of Glaubersalt, 1 per cent. of Alkali Blue, and 2 per cent. of borax. After boiling for 20 minutes, remove the skein, squeeze, rinse slightly (99), and pass into a fresh bath containing 300 cc. of water and 5 per cent. of sulphuric acid; enter at 160° F., bring to the boil and continue for 20 minutes (100). Notice that the full blue color of the dye is not developed until the material is treated with the acid bath. Borax is a mild alkali, and is added to the dye-bath for the purpose of insuring its remaining perfectly neutral. To show the effect of adding the acid directly to the dye-bath, prepare a bath containing 300 cc. of water, 10 per cent. of Glaubersalt, 1 per cent. of Alkali Blue, and 4 per cent. of sulphuric acid. Dye a skein of woolen yarn in this bath in the usual manner, and wash and dry (101). Compare the result with that obtained in the first method. Also notice that the addition of the acid to the bath causes the precipitation of the coloring-matter.



## NOTES.

1. **Preparation of the Dye-bath.** — For the dyeing of woolen yarn there will be required about 60 times the amount of water or dye liquor as there is material to be dyed; that is to say, 1 pound of woolen yarn will require about 7.5 gallons of water in the dye-bath (1 gallon of water weighs  $8\frac{1}{2}$  pounds). Silk will also require about the same proportion. Cotton yarn, on the other hand, will require only about one-half the amount of dye liquor as wool; that is to say, 1 pound of cotton may be dyed in about  $3\frac{1}{2}$  gallons of dye liquor. The porcelain beakers employed for the dye-tests should conveniently hold about 300 cc. of water; consequently they are of a convenient size for the dyeing of 5-gram skeins of woolen yarn or 10-gram skeins of cotton yarn, so that the woolen and cotton skeins employed for the tests should be supplied in these weights respectively. For silk, skeins of about 2 grams weight may be employed (for economy), consequently for such tests only about 125 cc. of dye liquor should be used.

2. **Calculations Used in Dyeing.** — The amounts of dyestuffs and chemicals employed in dyeing are usually expressed in terms of percentages on the weight of the material to be dyed. Thus in Exp. 36, "20 per cent. of Glaubersalt, 4 per cent. of sulphuric acid, and 1 per cent. of Acid Magenta" are called for; this would mean that the actual amounts of the substances to be taken are to be the respective percentages on the weight of the yarn dyed, which in this case is 5 grams. Therefore we have:

$$5 \text{ grams} \times .04 = 0.20 \text{ gram for the sulphuric acid,}$$

$$5 \text{ grams} \times .20 = 1.00 \text{ gram for the Glaubersalt,}$$

$$5 \text{ grams} \times .01 = 0.05 \text{ gram for the dyestuff.}$$

As these are rather small amounts to be continually weighing out, it is best to have the chemicals and dyestuffs employed in the dye-tests made up in solutions of such strength that convenient volumes will contain the required amounts as needed for the preparation of the dye-baths. As 4 per



cent. is the customary amount of sulphuric acid to employ, a convenient solution would be one of such strength that 10 cc. would contain 4 per cent. of the acid on 5 grams (this being the weight of the woolen yarn employed in all the tests). To prepare one litre or 1000 cc. of this solution, proceed as follows:

10 cc. is to contain 4 per cent. on 5 grams = 0.2 gram acid.

1000 cc. would therefore contain 20 grams acid.

As the commercial concentrated sulphuric acid has a density of about 1.84 (*i.e.*, 1 cc. weighs 1.84 grams), 20 grams by weight of the acid would be equivalent to  $20 \div 1.84 = 11.9$  cc. Hence the solution should contain 11.9 cc. of the concentrated sulphuric acid per litre. The solution of Glaubersalt may be prepared in a similar manner. A convenient strength is one containing 20 per cent. of the salt on 5 grams in 10 cc., which would be 1 gram in 10 cc., or 100 grams per litre. A convenient strength for the solutions of dyestuffs is 5 grams per litre; hence 1 per cent. on 5 grams would be equivalent to 10 cc. of the solution. For a 10-gram skein, where cotton yarn is employed in the tests, 20 cc. would be equivalent to 1 per cent.

**3. Function of the Chemicals Employed in Dyeing Acid Colors.** — These chemicals are mostly confined to *sulphuric acid* and *Glaubersalt*. As already explained under the general theory of dyeing, the sulphuric acid is used for the purpose of liberating the color-acid of the dyestuff so that it may more readily combine with the basic component of the fibre; hence the addition of the acid facilitates the dyeing and increases the exhaustion of the bath. Some acid dyes naturally require more acid than others to give the same degree of exhaustion, but 4 per cent. (on the weight of the wool) appears to be ample for almost all cases. The dyer does not vary the amount of acid with each individual dyestuff or variation in quantity of dyestuff used, but adheres to the fixed quantity of 4 per cent. Sometimes a better degree of exhaustion may be obtained by the addition of a further quantity (about 2 per cent.) of acid near the completion of the dyeing

operation. Some of the acid dyes do not require the addition of such a strong acid as sulphuric to liberate the color-acid, but give very good results with a milder acid, such as the organic acetic acid. Formic acid may also be used. Such colors are especially useful for the dyeing of wool-cotton fabrics, as the cotton is not injured by the acetic or formic acid. Nearly all of the acid colors are slightly dissociated on dissolving in water; that is to say, a small amount of the free color-acid is formed; hence a certain degree of dyeing takes place even without the addition of acid. Some of the acid dyes are largely dissociated in solution, and consequently these may be dyed in a neutral bath. Such colors are also useful for wool-cotton dyeing. Therefore the acid dyes may be divided into groups with reference to the required acidity of the dye-bath:

(a) Dyes which may be applied in a neutral bath.

(b) Dyes which require a slightly acid bath; usually acetic acid being employed.

(c) Dyes which require a bath acidulated with sulphuric acid.

The *glaubersalt* used in the dye-bath may exert an influence in several different ways:

(a) In mechanically retarding the interaction between the color-acid and the fibre.

(b) In chemically retarding the liberation of the color-acid from the dye-salt.

(c) In affecting the solubility of the dyestuff in the solution.

The first effect has been considered under the general theory of dyeing, and is very likely the chief influence exerted by the *glaubersalt*. There is a considerable possibility, however, that a chemical action may come into play. It is a well-established principle in chemistry that when one of the products of a chemical reaction is present in the solution, the rapidity and extent of this reaction will be reduced. Now, in the interaction between sulphuric acid and the dyestuff (which is generally the sodium salt of the color-acid) *glaubersalt* is formed; hence, if a relatively



large proportion of Glaubersalt is already present, the reaction between the acid and the dyestuff will be considerably retarded. Just to what extent this influence of the Glaubersalt affects the even dyeing of the acid colors is a question; but it is doubtful if it is as important as the preceding influence. As to the degree in which the Glaubersalt affects the solubility of the dyestuff in the solution, it may be stated that this must be rather limited, when it is considered that both the dyestuff and the Glaubersalt are in rather dilute solutions. The general effect of the addition of Glaubersalt to a dyestuff solution would be to lessen the solubility of the dye; but in order for this effect to be marked the concentration of the Glaubersalt in the solution would have to be rather high. If the Glaubersalt did act in this manner in the ordinary dye-bath, the effect would be to throw the dyestuff out of solution and hence to promote uneven rather than level dyeing; whereas we know the opposite to be the case. Furthermore, many of the acid dyes may be partially stripped from the fibre by boiling in a bath containing Glaubersalt; much more of the dyestuff being dissolved than if plain boiling water were used.

Glaubersalt is the common name for crystallized sodium sulphate; it has the chemical formula  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ . Calcined or desiccated Glaubersalt has the water of crystallization removed by heating; it is a white amorphous powder, having the formula  $\text{Na}_2\text{SO}_4$ . About 44 parts of desiccated Glaubersalt are equivalent to 100 parts of the crystalline salt.

**4. General Characteristics of the Acid Dyes.** — These colors are the principal dyes employed for the dyeing of woolen materials, especially yarns and piece-goods. Most of them are level-dyeing; and their general fastness to washing and light is good, though the fastness varies largely with the individual members. The acid dyes, as a rule, are cheap compared with the other classes of dyestuffs and considering their high coloring power. In this latter respect, however, they are not equal to the basic dyes, though these are more costly. In range of color the acid dyes are quite varied, representatives of almost every color being



available. Water of ordinary hardness does not have much effect on the acid dyes, and in the dye-bath any tendency of hard water to precipitate these colors is prevented by the presence of acid. Material dyed with the acid colors should be well washed after coming from the dye-bath, especially if heavy shades are used, otherwise the color may show unnecessary bleeding on scouring, or may have the defect of rubbing or "crocking." Should an acid color show any tendency towards dyeing unevenly, the following precautions should be observed:

(a) Start the dye-bath at a low temperature and heat to the boiling point only very gradually.

(b) Do not add any acid until the goods have been worked in the dye-bath for some time, and then add the acid in several portions during the dyeing.

(c) Be sure that the dyestuff is thoroughly dissolved and add the solution in several portions during the dyeing.

(d) Do not use too "short" a dye-bath, that is, one containing too little water.

The acid dyes, as a rule, exhaust fairly well, though this quality varies with individual members of the group. The exhaustion of the dye-bath may usually be considerably increased by the addition of extra acid towards the end of the dyeing operation. In case the bath does not exhaust well, it should be kept as a "standing kettle," that is, preserved for further use and freshened up by the addition of the necessary amount of dyestuff. Of course the poorer the exhaustion of the dye-bath the less will be the amount of the second addition of dyestuff. Generally speaking, with the third or fourth successive bath, the amount of dyestuff to be added each time will become constant in order to produce the same shade. Most of the acid dyes will give a "full shade" (in a starting bath) with about 3 to 4 per cent. of color. By a full shade is meant the maximum depth of color given by a dyestuff.

**5. Useful Data for Calculations in Dyeing.** — *To find the capacity of a rectangular tank:* Multiply the length by the breadth



by the depth (in feet), then multiply this product by 7.5 (the number of gallons in a cubic foot). The result will be the capacity of the tank in gallons.

*To find the capacity of a circular tank:* Find the square of half the diameter (in feet), multiply by  $\frac{2}{7}$  (an approximation to  $\pi = 3.1416$ ), then multiply by the depth (in feet), and finally multiply by 7.5. The result will be the capacity of the tank in gallons. A shorter approximation to the same result is as follows: square the diameter, multiply by the height, and then by the factor 5.9.

*To convert grams per litre into ounces per gallon:* Since one gallon is equivalent to  $3\frac{3}{4}$  litres, and one ounce is equal to 28.3 grams, multiply the number of grams per litre by  $3\frac{3}{4}$  and divide by 28.3. A briefer formula is to multiply grams per litre by the factor 0.133.

*To convert grams per kilogram into ounces per 100 pounds:* Multiply by the factor 1.6.

Grams per litre.	Grams per gallon.	Per gallon.			Grams per litre.	Grams per gallon.	Per gallon.		
		Lbs.	Ozs.	Grns.			Lbs.	Ozs.	Grns.
1	3.785			58	17	64.35		2	112
2	7.570			116	18	68.14		2	170
3	11.355			174	19	71.92		2	228
4	15.140			232	20	75.70		2	286
5	18.92			290	30	113.55		3	429
6	22.71			348	40	151.4		5	165
7	26.50			406	50	189.2		6	278
8	30.28		1	27	60	227.1		7	421
9	34.07		1	85	70	265.0		9	127
10	37.85		1	143	80	302.8		10	270
11	41.63		1	201	90	340.7		11	413
12	45.42		1	259	100	378.5		13	119
13	49.21		1	317	200	757.0	1	10	238
14	53.00		1	375	300	1135.5	2	7	357
15	56.76		1	433	400	1514.0	3	5	39
16	60.56		2	54	500	1892.0	4	2	158

EXAMPLE. — A solution of sulphuric acid of 1° Tw. density contains 8 grams of the acid per litre. Reference to the above table shows that this amount is equivalent to 1 oz. 27 grns. per gallon.

*To convert percentage of color into the corresponding quantity per 100 pounds of goods:*

Per cent.	Per 100 lbs.		Per cent.	Per 100 lbs.		Per cent.	Per 100 lbs.	
	Ozs.	Grns.		Ozs.	Grns.		Ozs.	Grns.
0.001		7	0.30	4	350	0.68	10	385
0.002		14	0.31	4	420	0.69	11	18
0.003		21	0.32	5	53	0.70	11	88
0.004		28	0.33	5	123	0.71	11	158
0.005		35	0.34	5	193	0.72	11	228
0.006		42	0.35	5	263	0.73	11	298
0.007		49	0.36	5	333	0.74	11	368
0.008		56	0.37	5	403	0.75	12	0
0.009		63	0.38	6	35	0.76	12	70
0.01		70	0.39	6	105	0.77	12	140
0.02		140	0.40	6	175	0.78	12	210
0.03		210	0.41	6	245	0.79	12	280
0.04		280	0.42	6	315	0.80	12	350
0.05		350	0.43	6	385	0.81	12	420
0.06		420	0.44	7	18	0.82	13	53
0.07	I	53	0.45	7	88	0.83	13	123
0.08	I	123	0.46	7	158	0.84	13	193
0.09	I	193	0.47	7	228	0.85	13	263
0.10	I	263	0.48	7	298	0.86	13	333
0.11	I	333	0.49	7	368	0.87	13	403
0.12	I	403	0.50	8	0	0.88	14	35
0.13	2	35	0.51	8	70	0.89	14	105
0.14	2	106	0.52	8	140	0.90	14	175
0.15	2	176	0.53	8	210	0.91	14	245
0.16	2	246	0.54	8	280	0.92	14	315
0.17	2	316	0.55	8	350	0.93	14	385
0.18	2	386	0.56	8	420	0.94	15	18
0.19	3	18	0.57	9	53	0.95	15	88
0.20	3	88	0.58	9	123	0.96	15	158
0.21	3	158	0.59	9	193	0.97	15	228
0.22	3	229	0.60	9	263	0.98	15	298
0.23	3	299	0.61	9	333	0.99	15	368
0.24	3	369	0.62	9	403	1.00	16	0
0.25	4	0	0.63	10	35	2.	32	0
0.26	4	70	0.64	10	105	3.	48	0
0.27	4	140	0.65	10	175	4.	64	0
0.28	4	210	0.66	10	245	5.	80	0
0.29	4	280	0.67	10	315	6.	96	0

EXAMPLE. — There is required 2.16 per cent. of dyestuff for 100 pounds of goods. Reference to the above table shows for 100 pounds of goods :

2 per cent. is 32 ozs.

0.16 per cent. is 2 ozs. 246 grns.

Hence

2.16 per cent. is 34 ozs. 246 grns.

*To convert cubic centimeters of test solutions containing one gram of dyestuff dissolved in one litre into corresponding percentages for 10-gram test-skeins and weights of dyestuff per 100 pounds of goods:*

Cc. of solution.	Per cent. on 10 grams.	Weight per 100 lbs.			Cc. of solution.	Per cent. on 10 grams.	Weight per 100 lbs.			Cc. of solution.	Per cent. on 10 grams.	Weight per 100 lbs.		
		Lbs.	Ozs.	Grns.			Lbs.	Ozs.	Grns.			Lbs.	Ozs.	Grns.
1	0.01			70	46	0.46	7	157	91	0.91		14		245
2	0.02			140	47	0.47	7	227	92	0.92		14		315
3	0.03			210	48	0.48	7	297	93	0.93		14		385
4	0.04			280	49	0.49	7	367	94	0.94		15		17
5	0.05			350	50	0.50	8	0	95	0.95		15		87
6	0.06			420	51	0.51	8	70	96	0.96		15		157
7	0.07	1		52	52	0.52	8	140	97	0.97		15		227
8	0.08	1		122	53	0.53	8	210	98	0.98		15		297
9	0.09	1		192	54	0.54	8	280	99	0.99		15		367
10	0.10	1		262	55	0.55	8	350	100	1.00	1	0		0
11	0.11	1		332	56	0.56	8	420	101	1.01	1	0		70
12	0.12	1		402	57	0.57	9	52	102	1.02	1	0		140
13	0.13	2		35	58	0.58	9	122	103	1.03	1	0		210
14	0.14	2		105	59	0.59	9	192	104	1.04	1	0		280
15	0.15	2		175	60	0.60	9	262	105	1.05	1	0		350
16	0.16	2		245	61	0.61	9	332	106	1.06	1	0		420
17	0.17	2		315	62	0.62	9	402	107	1.07	1	1		52
18	0.18	2		385	63	0.63	10	35	108	1.08	1	1		122
19	0.19	3		17	64	0.64	10	105	109	1.09	1	1		192
20	0.20	3		87	65	0.65	10	175	110	1.10	1	1		262
21	0.21	3		157	66	0.66	10	245	111	1.11	1	1		332
22	0.22	3		227	67	0.67	10	315	112	1.12	1	1		402
23	0.23	3		297	68	0.68	10	385	113	1.13	1	2		35
24	0.24	3		367	69	0.69	11	17	114	1.14	1	2		105
25	0.25	4		0	70	0.70	11	87	115	1.15	1	2		175
26	0.26	4		70	71	0.71	11	157	116	1.16	1	2		245
27	0.27	4		140	72	0.72	11	227	117	1.17	1	2		315
28	0.28	4		210	73	0.73	11	297	118	1.18	1	2		385
29	0.29	4		280	74	0.74	11	367	119	1.19	1	3		17
30	0.30	4		350	75	0.75	12	0	120	1.20	1	3		87
31	0.31	4		420	76	0.76	12	70	121	1.21	1	3		157
32	0.32	5		52	77	0.77	12	140	122	1.22	1	3		227
33	0.33	5		122	78	0.78	12	210	123	1.23	1	3		297
34	0.34	5		192	79	0.79	12	280	124	1.24	1	3		367
35	0.35	5		262	80	0.80	12	350	125	1.25	1	4		0
36	0.36	5		332	81	0.81	12	420	126	1.26	1	4		70
37	0.37	5		402	82	0.82	13	52	127	1.27	1	4		140
38	0.38	6		35	83	0.83	13	122	128	1.28	1	4		210
39	0.39	6		105	84	0.84	13	192	129	1.29	1	4		280
40	0.40	6		175	85	0.85	13	262	130	1.30	1	4		350
41	0.41	6		245	86	0.86	13	332	131	1.31	1	4		420
42	0.42	6		315	87	0.87	13	402	132	1.32	1	5		52
43	0.43	6		385	88	0.88	14	35	133	1.33	1	5		122
44	0.44	7		17	89	0.89	14	105	134	1.34	1	5		192
45	0.45	7		87	90	0.90	14	175	135	1.35	1	5		262

Cc. of solution.	Per cent. on 10 grams.	Weight per 100 lbs.			Cc. of solution.	Per cent. on 10 grams.	Weight per 100 lbs.			Cc. of solution.	Per cent. on 10 grams.	Weight per 100 lbs.		
		Lbs.	Ozs.	Grns.			Lbs.	Ozs.	Grns.			Lbs.	Ozs.	Grns.
136	1.36	1	5	332	158	1.58	1	9	122	180	1.80	1	12	350
137	1.37	1	5	402	159	1.59	1	9	192	181	1.81	1	12	420
138	1.38	1	6	35	160	1.60	1	9	262	182	1.82	1	13	52
139	1.39	1	6	105	161	1.61	1	9	332	183	1.83	1	13	122
140	1.40	1	6	175	162	1.62	1	9	402	184	1.84	1	13	192
141	1.41	1	6	245	163	1.63	1	10	35	185	1.85	1	13	262
142	1.42	1	6	315	164	1.64	1	10	105	186	1.86	1	13	332
143	1.43	1	6	385	165	1.65	1	10	175	187	1.87	1	13	402
144	1.44	1	7	17	166	1.66	1	10	245	188	1.88	1	14	35
145	1.45	1	7	87	167	1.67	1	10	315	189	1.89	1	14	105
146	1.46	1	7	157	168	1.68	1	10	385	190	1.90	1	14	175
147	1.47	1	7	227	169	1.69	1	11	17	191	1.91	1	14	245
148	1.48	1	7	297	170	1.70	1	11	87	192	1.92	1	14	315
149	1.49	1	7	367	171	1.71	1	11	157	193	1.93	1	14	385
150	1.50	1	8	0	172	1.72	1	11	227	194	1.94	1	15	17
151	1.51	1	8	70	173	1.73	1	11	297	195	1.95	1	15	87
152	1.52	1	8	140	174	1.74	1	11	367	196	1.96	1	15	157
153	1.53	1	8	210	175	1.75	1	12	0	197	1.97	1	15	227
154	1.54	1	8	280	176	1.76	1	12	70	198	1.98	1	15	297
155	1.55	1	8	350	177	1.77	1	12	140	199	1.99	1	15	367
156	1.56	1	8	420	178	1.78	1	12	210	200	2.00	2	0	0
157	1.57	1	9	52	179	1.79	1	12	280	300	3.00	3	0	0

EXAMPLE. — To obtain 0.67 per cent. of dyestuff on a 10-gram test-skein of wool or cotton, when the solution contains 1 gram of dyestuff per litre, it would be necessary to take 67 cc. of the solution; and for the dyeing of 100 pounds of goods this would be equivalent to 10 ounces 315 grains of dyestuff.

In case a 5-gram test-skein is used, the above figures in the percentage and weight columns are to be multiplied by 2. For instance, 1.34 per cent. of dyestuff would be equivalent to 67 cc. of the solution, or to 1 pound 5 ounces 193 grains on 100 pounds of material.

In case the dyestuff solution contains more than 1 gram per litre, it will be necessary to multiply the figures in the percentage and weight columns by the number of grams per litre of the solution. For instance, if the solution contains 5 grams per litre, 67 cc. would be equivalent to 3.35 per cent., or to 3 pounds 5 ounces 262 grains on 100 pounds, if a 10-gram test-skein is used.



If a 5-gram test-skein be used, 67 cc. of such a solution would be equivalent to 6.70 per cent., or to 6 pounds 11 ounces 87 grains on 100 pounds of goods.

*To convert percentage of color (on 100 pounds of goods) into quantity of standard solution of 4 ounces of dry color per gallon:*

Weight. Lbs.	Per cent.	Solution of 4 ozs. per gallon.			
		Quarts.	Pints.	Gills.	Noggins.
100	$\frac{1}{10}$	1	1	.....	$1\frac{6}{10}$
100	$\frac{1}{8}$	2	.....	.....	.....
100	$\frac{1}{6}$	3	.....	1	$1\frac{1}{2}$
100	$\frac{1}{4}$	4	.....	.....	.....
100	$\frac{1}{2}$	8	.....	.....	.....
100	1	16	.....	.....	.....

2 noggins = 1 gill

2 pints = 1 quart

4 gills = 1 pint

4 quarts = 1 gallon

*Convenient data regarding quantities of water based on United States gallon:*

1 gallon =	231.	cubic inches =	8.3	pounds	
1 quart =	57.75	" "	= 2.1	"	
1 pint =	28.88	" "	= 1.0	"	
1 gill =	7.22	" "	= .25	"	
1 cubic foot =	1728	" "	= 62.5	"	= 7.5 gallons
1 cubic inch			= .036	"	

#### *Comparative Strength of Different Chemicals.*

100 parts by weight of *sal soda* ( $\text{Na}_2\text{CO}_3 \cdot 10 \text{H}_2\text{O}$ ) is equivalent to about 37 parts of *soda ash* ( $\text{Na}_2\text{CO}_3$ ).

100 parts of *soda ash* is equivalent to 270 parts *sal soda*.

100 parts *crystallized glauber salt* ( $\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$ ) is equivalent to 44 parts of *calcined glauber salt* ( $\text{Na}_2\text{SO}_4$ ).

100 parts of *calcined glauber salt* is equivalent to 227 parts of *crystallized glauber salt*.

100 parts of *alum* (10.76 per cent.  $\text{Al}_2\text{O}_3$ ) has the same practical value as 60 parts of *aluminium sulphate* (18 per cent.  $\text{Al}_2\text{O}_3$ ).

100 parts of *aluminium sulphate* are equivalent to 170 parts *alum*.

100 parts of *sulphuric acid* (168° Tw.) correspond to 220 parts of *hydrochloric acid* (32° Tw.) or to 400 parts of *acetic acid* (9° Tw.).

100 parts of *hydrochloric acid* correspond to 45 parts of *sulphuric acid* or to 175 parts of *acetic acid*.

100 parts of *acetic acid* correspond to 26 parts of *sulphuric acid* or to 57 parts of *hydrochloric acid*.

### SAMPLES.

87. Representing the application of acid dyes to wool.
88. Dyed without glaubersalt.
89. Dyed with glaubersalt.
90. Dyed with 1 per cent. sulphuric acid.
91. Dyed with 2 per cent. sulphuric acid.
92. Dyed with 4 per cent. sulphuric acid.
93. Dyed with 8 per cent. sulphuric acid.
94. First dyeing from bath.
95. Second dyeing from bath.
96. Third dyeing from bath.
97. Dyed in a neutral bath.
98. Dyed with acid in bath.
99. Alkali blue before acid treatment.
100. Alkali blue after treatment with acid.
101. Alkali blue dyed with acid in the bath.

### QUIZ 6.

131. Outline briefly the general method of dyeing acid colors on wool.
132. Explain the function of the acid and the glaubersalt in the dye-bath.
133. What conditions of temperature should be maintained in dyeing? Explain the influence of the temperature of the bath on the dyeing process.
134. About what length of time is required for the dyeing to be completed? What precautions should be taken in working the material in the dye-bath to prevent felting?
135. Why should the material after dyeing be well washed? Does hard water have much influence in the dyeing of acid colors?
136. Are the acid colors much employed for the dyeing of wool? Give the general characteristics of the acid colors with respect to level dyeing, general fastness, cheapness, and range of colors.
137. Discuss the various precautions to be adopted for the production of even colors in the dyeing with acid dyes.

138. Explain the chemical and physical action of the glauber salt in the dye-bath in promoting even colors and good penetration.

139. What is glauber salt? What is meant by desiccated glauber salt? What quantity of glauber salt is usually employed in dyeing?

140. From your test to show the influence of the amount of acid in the dye-bath on the quality of the resulting color, what would you conclude as to the proper amount of acid to be employed?

141. What is meant by the "exhaustion" of the dye-bath? Do the acid dye-baths, as a rule, exhaust well? How may the relative exhaustion of the dye-bath be shown?

142. What is meant by a "standing kettle"? What influence does the exhaustion of the dye-bath have on the amount of dyestuff to be added for successive dyeings from the same kettle?

143. In what manner may the exhaustion of the dye-bath be increased? What is meant by a "full shade" of a dyestuff? What percentage of acid dye is usually necessary to give a full shade in the first bath?

144. Explain why some of the acid dyes may be applied to wool in a neutral bath. Is the exhaustion good in such cases? Under what circumstances would material be likely to be dyed with acid colors in a neutral bath?

145. How does the application of Alkali Blue differ from that of other acid dyes? Why cannot the acid be added directly to the dye-bath?

146. What is the purpose of adding borax to the dye-bath when Alkali Blue is used? What is borax? What is its chemical character?

147. Why is it necessary to after-treat Alkali Blue with an acid bath? What color is the material after dyeing in the first bath, and what color does it become when passed into the acid bath?

148. What is the effect of dyeing Alkali Blue with acid added directly to the dye-bath? Are there many acid dyes which require to be dyed in the manner of Alkali Blue?

149. What proportion should exist between the amount of woolen yarn to be dyed and the amount of dye liquor used? What proportion for silk? What proportion for cotton?

150. How many gallons of water are required for the dyeing of 1 pound of woolen yarn? How many pounds of water are in the United States gallon? In the English gallon?

151. Suppose it is required to dye 100 pounds of woolen yarn, and the dye-vat is 3 feet deep by 2 feet wide; how long would it have to be to contain the necessary dye liquor? (1 cubic foot of water weighs 62.5 pounds.)

152. What weights of woolen and cotton test-skeins may be conveniently dyed in a beaker holding 300 cc. of water? How much water would be required for the dyeing of test-skeins of silk weighing 2 grams?

153. In what manner are the amounts of dyestuffs and chemicals calculated in the preparation of dye-baths?



154. If 100 pounds of woolen yarn were to be dyed by the formula given in Exp. 36, what weights of Glaubersalt, acid, and dyestuff would be required? How many pints of sulphuric acid would be required? (1 pint of water is equivalent to 1 pound.)

155. If 56 pounds of woolen yarn are to be dyed, using the following amounts of dyestuffs:

- 0.75 per cent. Acid Magenta,
- 1.12 per cent. Orange ENZ,
- 0.08 per cent. Formyl Violet,

calculate the quantities of these dyes in pounds, ounces, and grains which would be required. Calculate the quantities in terms of grains. (See Appendix for tables of measures and weights.)

156. How would you prepare a solution of sulphuric acid of such strength that 10 cc. would be equivalent to 4 per cent. on 5 grams? How would you prepare such a solution that 5 cc. would be equivalent to 6 per cent. on 8 grams?

157. If a solution of dyestuff contains 5 grams of dry color per litre, how many cubic centimeters would be equivalent to 3.5 per cent. on 5 grams? To 2.75 per cent. on 10 grams?

158. If a 2-gram skein of silk is employed for a dye-test the dye solution should contain how many grams per litre in order that 10 cc. may be equivalent to 1 per cent. in dyeing the silk?

159. If a solution of dyestuff is used containing 4 ounces per gallon, how much would be required for 1 per cent. on 10 pounds of yarn? For one-quarter per cent. on 25 pounds? For  $2\frac{1}{4}$  per cent. on 45 pounds? (Give the result in terms of gallons, quarts, pints, gills, and noggins.)

160. How many grams of dyestuff would be needed to give 0.08 per cent. on 125 kilos? For 1.25 per cent. on 25 kilos? For one-quarter per cent. on 10 kilos?

161. How many grams of dyestuff would be required to give 0.05 per cent. on 10 pounds of yarn? For 0.75 per cent. on 5 pounds? For  $2\frac{1}{8}$  per cent. on 3 pounds?

162. How much borax would be required to give a litre of a solution such that 10 cc. would be equivalent to 2 per cent. on 5 grams? How many cubic centimeters of a borax solution containing 50 grams per litre would be needed to give 2 per cent. on 5 grams?

163. 100 grams per 100 kilos is equivalent to how many ounces per 100 pounds? 10 grams per kilo is equivalent to how many grains per pound? 2 ounces per 100 pounds is equivalent to how many grams per 100 kilos? 1 ounce 53 grains per pound is equivalent to how many grams per kilo?

164. If a 5-gram sample were dyed with 0.2 per cent. of color, how much (in ounces and grains) would be needed to obtain the same color on 100 pounds of material?



## SECTION VII.

### APPLICATION OF ACID DYES.

**Experiment 42. Dyeing Acid Dyes on Acidified Wool.** — Wool combines with acids with considerable affinity, and when so treated will dye with the acid colors without any further addition of acid to the dye-bath. Carbonized shoddy (recovered wool fibre treated with acid for the purpose of decomposing vegetable fibres), on this account, will generally dye more heavily than ordinary wool under the same conditions. Work a test-skein of woollen yarn in a bath containing 300 cc. of water and 10 per cent. of sulphuric acid, boiling for 15 minutes. Then rinse in fresh water and squeeze. Dye this skein, together with one of ordinary wool, in a bath containing 300 cc. of water, 20 per cent. of glaubersalt, and 1 per cent. of Formyl Violet 10 B. After dyeing wash well and dry (102, 103). Then compare the two skeins for depth of color, and it will be found that the one treated with acid has been dyed much the deeper shade.

**Experiment 43. After-treatment of an Acid Dye with Chrome.** — Some of the acid dyes on being treated after dyeing with a boiling solution of chrome (potassium bichromate) are changed into faster and deeper colors. The chrome may act in two ways; in the first place, it may combine with the dyestuff to give a permanent color-lake (similar to the mordant dyes), and secondly, it may cause an oxidation of the dyestuff whereby a new compound is obtained on the fibre which is faster in color than the original one. Dye two skeins of woollen yarn in the usual manner in a bath containing 300 cc. of water, 10 per cent. of glaubersalt, 4 per cent. of sulphuric acid, and 1 per cent. of Cloth Red GA. After dyeing for one-half hour, lift the skeins from the bath (104) and add 2 per cent. of chrome. Reënter one of the skeins, and continue boiling for 20 minutes. Then wash and dry (105).

Compare the color of the two skeins, and it will be noticed that the chromed one is deeper in shade. Test the fastness of the dyeings to washing in the following manner: Take a portion of each skein and plait it with some strands of white woolen yarn, and then scour the samples so prepared in a lukewarm dilute soap solution containing about 5 grams of soap per litre. Next wash in fresh water, and allow the two tests (106, 107) to dry, and compare them as to the loss of color and as to the amount of color that bleeds into the white wool.

**Experiment 44. Use of Acetic Acid in Dyeing Acid Colors.** — Some of the acid dyes tend to go on to the fibre too rapidly if sulphuric acid is used in the bath, owing to the fact that the color acid is liberated too rapidly and has a strong affinity for the fibre; hence uneven dyeings are liable to result. This fault may be avoided by using a weak acid, such as acetic acid, and not adding all of the acid at once but in several portions. To more thoroughly exhaust the bath some sulphuric acid may be added towards the end of the dyeing operation. Prepare a dye-bath containing 300 cc. of water, 20 per cent. of Glaubersalt, and 1 per cent. Alizarin Lanacyl Blue, and dye a skein of woolen yarn as usual for 20 minutes, then lift the skein (108) and add 2 per cent. of acetic acid, and continue the dyeing for 10 minutes (109). Lift the skein a second time and add 2 per cent. of sulphuric acid and continue dyeing for 15 minutes. Then wash and dry (110).

**Experiment 45. Use of a Chromotrop Dye.** — This class of dyes gives red or brown colors when dyed in an acid bath, but the color so obtained is of little importance. When after-treated, however, with solutions of metallic salts (such as chrome) the color changes to black and becomes very fast. Prepare a bath containing 300 cc. of water, 4 per cent. of sulphuric acid, 20 per cent. of Glaubersalt, and 6 per cent. of Chromotrop FB; dye two skeins of woolen yarn in this bath in the usual manner for one-half hour, then lift (111) and add 3 per cent. of chrome and 2 per cent. of sulphuric acid, reënter one of the skeins and continue boiling for 20 minutes (112).

**Experiment 46. Use of Phthalein Dyes.**—These dyes are represented by the eosins and related coloring-matters. They are applied in neutral or weakly acid baths, and give delicate red and pink shades which are characterized by a peculiar brightness and fluorescence. The shades may also be made more brilliant by dyeing on wool which has first been treated with alum. Prepare a bath containing 300 cc. of water, 10 per cent. of Glaubersalt, and 1 per cent. of Eosin, and dye a skein of woolen yarn in the usual manner (113). Prepare a second bath containing 300 cc. of water, 10 per cent. of Glaubersalt, 5 per cent. of acetic acid, and 1 per cent. of Eosin, and dye a skein of woolen yarn in the usual manner (114). Prepare a third bath containing 300 cc. of water, 5 per cent. of alum and 5 per cent. of tartar, and 5 per cent. of acetic acid; boil a skein of woolen yarn in this bath for one-half hour, then lift and add 1 per cent. of Eosin, and continue dyeing for 20 minutes (115). The Erythrosine, Phloxine, and Rose Bengale also belong to this group of phthalein dyes. The tartar is used in the bath to aid in the decomposition of the alum.

**Experiment 47. General Method of Dyeing Acid Dyes on Cotton.**—Cotton has no direct affinity for the acid colors and requires a basic mordant to combine with the color-acid of the dyestuff. Alum is used at times for this purpose. Prepare a bath containing 250 cc. of water, 20 per cent. of alum, 20 per cent. of Glaubersalt, and 2 per cent. of Water Blue; enter a skein of cotton yarn at 140° F., raise to 180° F. and keep at that temperature for 45 minutes; then squeeze and dry without washing (116). It will be noticed that a rather concentrated or "short" bath is employed and that even then the exhaustion is very imperfect. These dyes are not much used on cotton at the present time, except for such materials as curtains, etc., where bright colors are desired which have good fastness to light and where fastness to washing is not demanded. To show the lack of fastness to washing of this color, plait a portion of the dyed sample with some strands of white cotton yarn, and scour this test sample in a dilute soap solution (117); it will be found that the color will wash out almost



completely. When applied to cotton, these dyes are usually known as "alum colors" because that salt is used in the bath.

**Experiment 48. Dyeing in a Neutral Salt Bath.** — This method is generally employed for the dyeing of bright pale shades on cotton with the acid dyes. Use a skein of bleached cotton yarn, and dye in a bath containing 250 cc. of water, 10 grams of common-salt, and 10 per cent. of Eosin; work for 45 minutes at a temperature of  $140^{\circ}\text{F.}$ , then squeeze and dry without washing (118). The large amount of salt employed helps to better exhaust the bath, as the dyestuff is less soluble in salt solutions. The bath, however, is in no wise exhausted and should be employed in practice for a "standing" bath for the dyeing of subsequent lots.

**Experiment 49. Use of "Blue Mordant."** — This mordant is a tartrate of aluminium, and may be prepared by dissolving 22 parts of aluminium sulphate in 45 parts of water, and then adding a solution of  $4\frac{1}{2}$  parts of tartaric acid dissolved in 20 parts of water, after which gradually add a solution of  $6\frac{1}{2}$  parts of soda ash in 35 parts of water, and dilute the whole to 175 parts with water. For mordanting, use 1 part of this solution to 30 parts of water, or 10 cc. to 300 cc. of water. Work a skein of cotton yarn in a bath containing 300 cc. of water, 10 cc. of "blue mordant," and 1 per cent. of Water Blue 6B for one-half hour at  $160^{\circ}\text{F.}$  Squeeze and dry without washing (119).

**Experiment 50. Use of Sodium Stannate Mordant.** — This salt is easily decomposed when its solution is boiled, and thus liberates oxide of tin in the fibre. It is used as follows: Steep a skein of cotton yarn for one-half hour in a bath containing 200 cc. of water and 5 grams of sodium stannate at  $180^{\circ}\text{F.}$  Remove the skein, squeeze, and dye in a bath containing 250 cc. of water and 1 per cent. of Ponceau 4B and 5 per cent. of alum; enter at  $140^{\circ}\text{F.}$ , gradually raise to  $190^{\circ}\text{F.}$ , and dye at that temperature for one-half hour. Squeeze and dry without washing (120).

**Experiment 51. Dyeing of Silk with Acid Dyes.** — Dye a test-skein of silk yarn in a bath containing 150 cc. of water,



2 per cent. of sulphuric acid, and 2 per cent. Naphthol Yellow; enter at  $120^{\circ}$  F., gradually raise to the boil, and dye at that temperature for one-half hour, then wash well and "lustre" by passing through a bath containing 1 gram of tartaric acid and 150 cc. of water at  $100^{\circ}$  F. Squeeze without washing and dry (121). Like wool, silk will also combine directly with the acid colors. Usually a bath is employed containing a considerable amount of boiled-off liquor acidified with acetic acid. This is to prevent as little loss in the weight of the silk as possible during the dyeing, as silk usually comes to the dyer still containing more or less of the silk glue, which would come off in the dye-bath if there were not a considerable amount of the same substance present.

**Experiment 52. Use of Acetic Acid in Dyeing Silk.** — Dye a test-skein of silk yarn in a bath containing 150 cc. of water, 4 per cent. of acetic acid, and 2 per cent. Eosin; enter at  $120^{\circ}$  F., gradually raise to the boil, and dye at this temperature for one-half hour. Then wash well and brighten as in Exp. 51. Squeeze and dry (122). This method of dyeing is used where feebly acid dyes are employed.

**Experiment 53. Use of Boiled-off Liquor in Dyeing Silk.** — Boiled-off liquor is the scouring bath left after the scouring of silk with strong soap solutions, and consists of the solution of soap and silk-glue. Prepare a bath containing 15 cc. of boiled-off liquor and 125 cc. of water, 2 per cent. of Brilliant Croceine, and sufficient sulphuric acid to give the bath a decidedly acid reaction with litmus paper. The presence of the silk-glue prevents the precipitation of the soap by the addition of the acid. Dye a test-skein of silk yarn in this bath, entering at  $100^{\circ}$  F. and gradually raising to  $180^{\circ}$  F., and continue at that temperature for one-half hour. Wash well and brighten as described in Exp. 51. Squeeze and dry (123).

## NOTES.

1. **List of the Principal Acid Dyestuffs.** — The acid dyes to be met with upon the market at the present time include a very large number. This is further increased by the fact that many different names are frequently given to the same dyestuff by different manufacturers and dealers, and still further by the use of mixed dyes to produce different shades and tones. It would be practically impossible to give a complete list of all the acid dyes which are sold, but the following will give a fair idea of the principal dyes. They are roughly classified according to color.

## (a) RED.

Acid Carmoisine.	Azo Grenadine.
Acid Cerise.	Azo Phloxine.
Acid Fuchsine.	Azo Orseille.
Acid Magenta.	Azo Red.
Acid Maroon.	Azo Rubine.
Acid Red.	Benzyl Red.
Acid Rhodamine.	Biebrich Acid Red.
Acid Rosamine.	Biebrich Scarlet.
Acid Ponceau.	Bordeaux.
Alkali Fast Red.	Brilliant Acid Carmine.
Amaranth.	Brilliant Bordeaux.
Amido Naphthol Red.	Brilliant Carmoisine.
Anisoline.	Brilliant Cochineal.
Anthracene Red.	Brilliant Croceine.
Apollo Red.	Brilliant Double Scarlet.
Archil Substitute.	Brilliant Fast Red.
Azo Acid Carmine.	Brilliant Orseille.
Azo Acid Fuchsine.	Brilliant Ponceau.
Azo Acid Rubine.	Brilliant Rubine.
Azo Acid Magenta.	Brilliant Scarlet.
Azo Bordeaux.	Brilliant Sulphon Red.
Azo Cardinal.	Cardinal.
Azo Carmine.	Cardinal Red.
Azo Coccine.	Carmoisine.
Azo Cochineal.	Cerasine.
Azo Crimson.	Chromazon Red.
Azo Eosin.	Chromotrop.
Azo Fuchsine.	Chromotrop 2R.

## (a) RED. — Continued.

Clayton Cloth Red.	Milling Red.
Cloth Red.	Milling Scarlet.
Cloth Scarlet.	Naphthorubine.
Coccine.	Naphthol Red.
Coccinine.	Naphthol Scarlet.
Cochineal Red.	Naphthylamine Red.
Cochineal Scarlet.	New Claret.
Cotton Scarlet.	New Coccine.
Cresol Red.	New Red.
Croceine.	Orcelline.
Croceine Scarlet.	Orseille Red.
Crystal Ponceau.	Palatine Red.
Cyanosine.	Palatine Scarlet.
Double Brilliant Scarlet.	Phloxine.
Double Ponceau.	Ponceau.
Double Scarlet.	Pyrotine Red.
Emin Red.	Roccelline.
Eosamine.	Rock Scarlet.
Eosin.	Rosazeine.
Eosin Scarlet.	Rose Bengale.
Erythrine.	Rosinduline.
Erythrosine.	Roxamine.
Fast Acid Eosin.	Salicine Red.
Fast Acid Fuchsine.	Scarlet.
Fast Acid Phloxine.	Silk Red.
Fast Bordeaux.	Silk Scarlet.
Fast Claret Red.	Sorbine Red.
Fast Ponceau.	Sulphon Carmine.
Fast Red.	Tolane Red.
Fast Scarlet.	Tyemond Red.
Florida Red.	Tyemond Scarlet.
Guinea Bordeaux.	Victoria Rubine.
Guinea Carmine.	Victoria Scarlet.
Lanafuchsine.	Violamine.
Mars Red.	Wool Red.
Mercerine Wool Red.	Wool Scarlet.
Mercerine Wool Scarlet.	

## (b) ORANGE.

Aniline Orange.	Milling Orange.
Aurantia.	Orange I.
Brilliant Orange.	Orange II.
Croceine Orange.	Orange IV.
Crystal Orange.	Orange R, G, etc.
Gold Orange.	Palatine Orange.
Kermesine Orange.	Pyrotine Orange.
Mandarin G.	Tyemond Orange.

## (c) YELLOW.

Acid Yellow.	Mercerol Wool Yellow.
Alkali Yellow.	Metanil Yellow.
Alpine Yellow.	Milling Yellow.
Azo Acid Yellow.	Naphthol Yellow.
Azo Flavine.	Naphthol Yellow S.
Azo Yellow.	Naphthylamine Yellow.
Brilliant Yellow.	New Yellow.
Chinoline Yellow.	Persian Yellow.
Chrysoine.	Picric Acid.
Cinereine.	Quinoline Yellow.
Citronine.	Resorcine Yellow.
Curcumine.	Solid Yellow.
Fast Yellow.	Sun Yellow.
Fast Light Yellow.	Tartrazine.
Flavaniline.	Tropæoline.
Flavazine.	Tyemond Yellow.
Golden Yellow.	Uranine.
Helianthine.	Victoria Yellow.
Indian Yellow.	Wool Yellow.
Martius Yellow.	Xanthamine.

## (d) GREEN.

Acid Green.	Brilliant Milling Green.
Alkali Fast Green.	Cyanole Green.
Alizarin Green.	Cyprus Green.
Alizarin Cyanine Green.	Diamond Green.
Anthracene Acid Green.	Domingo Green.
Benzyl Green.	Eboli Green.
Brilliant Acid Green.	Fast Acid Green.



## (d) GREEN. — Continued.

Fast Green.	Naphthaline Green.
Fast Green Bluish.	Naphthol Green.
Fast Light Green.	Neptune Green.
Guinea Green.	Night Green.
Kiton Green.	Patent Green.
Light Green.	Wool Green.
Milling Green.	

## (e) BLUE.

Acid Peacock Blue.	Fast Blue Black.
Alizarin Blue SAP, SAE.	Fast Blue for Wool.
Alizarin Pure Blue.	Fast Sky Blue.
Alizarin Saphirol.	Fluorescent Blue.
Alkali Blue.	Formyl Blue.
Anthra Cyanine.	Full Blue.
Anthracene Blue.	Gallanyl Indigo.
Azine Blue.	Gallocyanine.
Azo Acid Blue.	Gallazin A.
Azo Marine Blue.	Gentiana Blue.
Bavarian Blue.	Indigo Blue.
Benzyl Blue.	Indigo Carmine.
Biebrich Acid Blue.	Indigo Extract.
Blackley Blue.	Indigo Substitute.
Brilliant Blue.	Indigotine.
Brilliant Silk Blue.	Indocyanine.
Carmine Blue.	Induline.
China Blue.	Intensive Blue.
Cloth Blue.	Ketone Blue.
Coomassie Navy Blue.	Kiton Blue.
Copper Blue.	Lanacyl Blue.
Cotton Blue.	Lanacyl Marine Blue.
Cyanine.	Lazuline Blue.
Cyanole.	Lyons Blue.
Cyprus Blue.	Marinol Acid Blue.
Disulphine Blue.	Marine Blue.
Eriochlorine.	Methane Dark Blue.
Eriocyanine.	Methyl Alkali Blue.
Erioglaurine.	Methyl Soluble Blue.
Fast Acid Blue.	Milling Blue.
Fast Blue.	Naphthaline Blue.

## (e) BLUE. — Continued.

Naphthazine Blue.	Sapphire Blue.
Naphthol Blue.	Silk Blue.
Naphthyl Blue.	Solid Blue.
Navy Blue.	Soluble Blue.
New Patent Blue.	Spirit Blue.
Night Blue.	Sulphon Acid Blue.
Opal Blue.	Thiocarmine.
Patent Blue.	Urania Blue.
Patent Marine Blue.	Victoria Marine Blue.
Patent Neutral Blue.	Water Blue.
Peri Wool Blue.	Wool Blue.
Pure Blue.	Wool Marine Blue.

## (f) VIOLET.

Acid Mauve.	Formyl Violet.
Acid Violet.	Guinea Violet.
Alkali Violet.	Lanacyl Violet.
Azo Acid Violet.	Naphthyl Violet.
Benzal Violet.	Neutral Violet.
Benzyl Violet.	Red Violet.
Biebrich Acid Violet.	Regina Violet.
Fast Acid Violet.	Victoria Violet.
Fast Sulphon Violet.	Violamine.
Fast Violet.	Wool Violet.

## (g) BROWN.

Acid Brown.	Dark Acid Brown.
Azo Brown.	Fast Brown.
Bismarck Acid Brown.	Marron.
Bronze Acid Brown.	Naphthol Brown.
Chestnut Brown.	Naphthylamine Brown.
Chromogen.	Resorcin Brown.
Clayton Wool Brown.	

## (h) BLACK.

Acid Black.	Anthracene Acid Black.
Alizarin Black.	Anthracite Black.
Amido Naphthol Black.	Azo Acid Black.
Aniline Gray.	Azo Black.

## (h) BLACK. — Continued.

Azo Merino Black.	Naphthyl Blue Black.
Biebrich Patent Black.	Naphthylamine Black.
Brilliant Black.	Nerol.
Cashmere Black.	New Victoria Black.
Coomassie Black.	Nigrosine.
Copper Black.	Palatine Black.
Deep Black.	Patent Palatine Black.
Domingo Acid Black.	Phenol Black.
Domingo Azo Black.	Phenylamine Black.
Domingo Blue Black.	Phenylene Black.
Domingo Violet Black.	Silk Black.
Mercerol Wool Black.	Sudan Black.
Methane Black.	Victoria Black.
Naphtacyl Black.	Wool Black.
Naphthaline Acid Black.	Wool Deep Black.
Naphthol Black.	Wool Gray.

## SAMPLES.

102. Wool not acidified, dyed.
103. Acidified wool dyed.
104. Cloth Red before chroming.
105. Cloth Red after chroming.
106. Scouring test of unchromed sample.
107. Scouring test of chromed sample.
108. Alizarin Lanacyl Blue before adding acid.
109. After addition of acetic acid.
110. After addition of sulphuric acid.
111. Chromotrop dye before chroming.
112. Chromotrop dye after chroming.
113. Phthalein dye in neutral bath.
114. Phthalein dye in acetic acid bath.
115. Phthalein dye on wool treated with alum.
116. Water Blue on cotton with alum.
117. Scouring test with Water Blue.
118. Acid dye on cotton in neutral salt bath.
119. Use of "blue mordant" on cotton.
120. Use of sodium stannate mordant on cotton.
121. General method of dyeing silk with acid dyes.
122. Dyeing silk in an acetic acid bath.
123. Dyeing silk with acid dye in bath of boiled-off liquor.

## QUIZ 7.

165. Does wool combine chemically with acids? What proof can you give of this?

166. Does acidified wool react in the same manner as ordinary wool with acid colors? What is the reason of this?

167. What is meant by "carbonized" shoddy? Why does this dye up differently from ordinary wool?

168. What is chrome? What is its action on some acid dyes? What is the purpose of the after-treatment with chrome?

169. How is the after-treatment with chrome carried out? What effect has the treatment on the shade of Cloth Red?

170. Does the treatment with chrome have any effect on the fastness of Cloth Red to scouring? How was the scouring test conducted?

171. Why is acetic acid sometimes used in place of sulphuric acid in the dyeing of acid colors?

172. How is acetic acid applied in the acid dye-bath? Why is sulphuric acid often added towards the end of the dyeing?

173. How are the chromotrop dyes applied? Explain the action of metallic salts on the color.

174. What color does Chromotrop FB give before chroming? After chroming?

175. What is meant by a phthalein dye? What characteristic property do these dyes possess?

176. In what three ways may eosin be applied to wool? Compare the results of the processes.

177. Of what chemical character are the phthalein dyes? Explain the action of the alum on the dyestuff.

178. In the preparation of the wool with alum, what is the action of the tartar?

179. What other dyes belong to the phthalein class? What colors are these dyes?

180. How must cotton be prepared in order to dye with acid colors? What character of mordant must be used?

181. What mordant is mostly used for dyeing acid colors on cotton? How is the dyeing operation carried out?

182. What is meant by a "short" bath? Does the acid dye-bath with cotton exhaust well?

183. Are acid dyes much used for cotton dyeing? On what classes of cotton goods are they mostly used?

184. Are the acid dyes on cotton fast to light? To washing? What was the result of your washing test, and how was the test conducted?

185. What is meant by "alum" colors on cotton, and why are they so called?



186. Give the method of applying acid dyes to cotton in a neutral salt bath. For what character of colors is this method used?

187. Explain the action of the salt in the dye-bath with acid colors on cotton. What is meant by a "standing" bath, and what are the advantages of using standing baths in dyeing?

188. Of what does "blue mordant" consist? How is it prepared?

189. How is "blue mordant" employed in connection with acid colors on cotton? How do the results obtained by its use compare with those dyed in a plain alum bath?

190. What is the general method of dyeing acid colors on silk?

191. What is meant by "lustring" or "brightening" silk after dyeing, and how is this done? How does this treatment affect the "scroop" of the silk?

192. Under what conditions is acetic acid used in dyeing silk? Why is boiled-off liquor used in the dye-bath?

193. Give the method of dyeing acid colors on silk in a bath of boiled-off liquor. At what temperatures is the dyeing done?

## SECTION VIII.

### REPRESENTATIVE ACID DYES.

**Experiment 54. Representative Acid Dyes on Wool.** — Dye test-skeins of woolen yarn in baths containing 300 cc. of water, 20 per cent. of Glaubersalt, 4 per cent. of sulphuric acid, and 1 per cent. respectively of the following dyestuffs:

Naphthol Red EB (Cass.) (124). Acid Violet 5BF (Metz) (129).  
Emin Red (Ber.) (125). Patent Blue V (Metz) (130).  
Tartrazine (K. & P.) (126). Orange II (S. & S.) (131).  
Naphthol Yellow S (Sch.) (127). Wool Blue 2B (Ber.) (132).  
Alizarin Blue SAE (Elb.) (128). Acid Green (Elb.) (133).

Enter at 140° F., gradually raise to the boil, and continue at that temperature for one-half hour; wash well and dry. These test-skeins are to be preserved for the purpose of testing the colors for fastness to various agencies.

**Experiment 55. Representative Acid Dyes on Cotton.** — Dye test-skeins of cotton yarn in baths containing 200 cc. of water, 20 per cent. of alum, and 50 per cent. of common-salt at a temperature of 180° F. for 1 hour. Wring out and dry without washing. Preserve the dyed skeins for the purpose of testing the colors for fastness.

Use 10 per cent. of the following dyestuffs:

Brilliant Orange G (Metz) (134).  
Ponceau 4R (Ber.) (135).  
Rose Bengale (Metz) (136).  
Methyl Blue (Ber.) (137).  
Brilliant Croceine M (Cass.) (138).  
Metanil Yellow (Ber.) (139).  
Erythrosine B (Cass.) (140).  
Irisamine G (Cass.) (141).

**Experiment 56. Representative Acid Dyes on Silk.**—Dye test-skeins of silk yarn in baths containing 150 cc. of water, 15 cc. of boiled-off liquor, and acidify with sulphuric acid. Dye for 1 hour at 180° F. Wash well and brighten with tartaric acid.

Use 2 per cent. of the following dyestuffs:

Acid Magenta (R.H.) (142).	Crystal Ponceau 6R (Cass.)
Acid Violet 4RS (Metz) (143).	(147).
Methyl Blue for silk (Metz) (144).	Cyanole BB (Cass.) (148).
Orange II (S. & S.) (145).	Acid Green (Elb.) (149).
Lyons Blue (S. & S.) (146).	Azo Fuchsine (Elb.) (150).
Silk Black 4BF (Ber.) 6 per cent. (151).	

# NOTES.

**1. Dyestuff Manufacturers.**—The abbreviations after the names of the dyestuffs refer to the names of the manufacturers or American agents, as the case may be. The principal manufacturers of dyestuffs with their American agents are as follows:

*Bad.* for Badische Company (formerly Kuttroff, Pickhardt & Co.), agents for the Badische Anilin und Soda Fabrik (B. A. S. F.) of Ludwigshafen, Germany.

*Elb.* for Farbenfabriken of Elberfeld Company, agents for the same company of Elberfeld, Germany.

*Ber.* for Berlin Aniline Works, agents for the Actien Gesellschaft für Anilin Fabrikation, of Berlin, Germany.

*Cass.* for Cassella Color Company, agents for Leopold Cassella & Co., of Frankfort, Germany.

*Metz* for H. A. Metz & Co., agents for the Farbwerke Höchst, vorms. Meister, Lucius, & Brunig Company, of Höchst, Germany.

*Kalle* for Kalle & Co., agents for the same company, of Biebrich, Germany.

*Sykes* for Walter F. Sykes & Co. (formerly Sykes & Street), agents for Société Anonyme des Matières Colorantes et Produits Chimiques de St. Denis, of France.

*Nat.* for National Aniline Company (formerly Hartford, Hanna & Schoellkopf Company), of Buffalo, American manufacturers of dyes.

*Bch.* for Beach & Co., agents for British Alizarin Company, England; for Brooke, Simpson, & Spiller, England; for Farbwerke Griesheim, Germany; and for Société Chimique des Usines de Rhône, France.

*Bd.* for J. A. & W. Bird Company, agents for the Clayton Aniline Company, England.

- Bs.* for C. Bischoff & Co., agents for Farbenfabriken Dahl & Co., Elberfeld, Germany, and for Farbwerk Mühlheim, vorms. A. Leonhardt & Co., Mühlheim, Germany.
- Klp.* for A. Klipstein & Co., agents for Farbwerke vorms. Durand, Huguenin & Co., Basle, Switzerland; for Gesellschaft für Chemische Industrie, of Basle, Switzerland; and for Carl Neuhaus, of Elberfeld, Germany.
- Kell.* for John J. Keller & Co., agents for Anilin und Extract Fabriken, of Basle, Switzerland.
- Lev.* for Levinstein, Slackle & Crumpsall, England.
- Gy.* for Geigy Aniline and Extract Company, agents for the same company, of Basle, Switzerland.
- Gei.* for Geisenheimer & Co., agents for K. Oehler, Offenbach, Germany.
- RH.* for Read Holliday & Sons, agents for the same company, of England.
- Ly.* for Thomas Leyland & Co., agents for Levinstein, England.
- AD.* for Andreykovicz & Dunck, agents for Chemische Fabriken, vorms. Weiler-ter-Meer, Uerdingen, Germany.

The letters after the names of dyestuffs are usually private trade distinctions for the use of the manufacturer in identifying the color. They may also refer to the particular shade of the dyestuff, as, for example, B stands for blue shade, R for a red shade, G (gelb) for a yellow shade, etc.

**2. On the Proper Storage of Dyestuffs.** — Dyestuffs should be kept in a cool dry room, and any barrels or tins which have been opened should be kept well covered up, otherwise one color may become contaminated by dust from another dye, and the dyestuffs are liable to absorb moisture from the air. The absorption of moisture may cause the dyestuff to cake together and become difficult to dissolve, and besides the dyestuff will alter its weight by the amount of moisture absorbed. Steam from the dyehouse should be carefully excluded from the drug room in which the dyes are stored. Dyestuffs in the form of pastes should always be well stirred up before weighing out and should be kept from exposure to the air, otherwise the water of the paste will evaporate and the dyestuff will alter very materially in its strength. Paste dyes are usually of 20 per cent. strength; that is, they contain 20 per cent. of actual dyestuff, the rest being water.

**3. On the Dissolving of Dyestuffs.** — Dyes should be dissolved by stirring up with a sufficient amount of pure boiling water.



Distilled water is the best to use for this purpose, but where this is not available, water from condensed steam should be employed. All dyes should be well dissolved before being added to the dye-bath. After dissolving, the dye solution should be filtered through a fine hair sieve or through a coarse cotton cloth, in order to prevent undissolved particles and lumps from passing into the bath. A few colors like Auramine should not be dissolved in boiling water, as the color is partially decomposed; such colors should be dissolved at about 160° to 180° F. The amount of water required for dissolving dyes varies greatly with the different colors; 250 parts of water to 1 part of dye will be sufficient for even such dyes as are soluble with difficulty; for readily soluble dyes from 10 to 50 parts of water will be sufficient. In general it may be said that for difficultly soluble dyes one-half pound may be dissolved in 10 gallons of water, and for ordinary dyes about 2 pounds may be dissolved in 10 gallons of water. Hard water should not be employed for the solution of dyestuffs, as many dyes are precipitated or otherwise affected by the metallic salts present in such water; this is especially true of the basic dyes. Where it is necessary to use hard water, it should be corrected by the addition of a small amount of acetic acid, or with soda, depending on the nature of the dyestuff. For basic dyes acetic acid should be used, sufficient being added to the water to give it an acid reaction with litmus paper. Any acid dyes which may be affected by the hardness of the water should also be dissolved with the aid of acetic acid. For Water Blue, Alkali Blue, or Acid Violet, acid should not be used, but a little soda or borax should be added to the water. The same is also true for the general class of phthalein dyes, in which case the hard water should be boiled up with a little soda ash, allowed to settle and used for dissolving the dyestuff. In dissolving substantive dyes, hard water should also be corrected with the addition of a small amount of soda. The precipitation of dyestuff from standing baths is generally caused by the use of impure water or by the gradual accumulation of too much salt in the bath.

**4. Apparatus for Dyeing.** — Yarn is mostly dyed in suitable wooden vats, the skeins being suspended in the vats from wooden sticks. Many dyes are more or less sensitive to the action of metals, though in many cases bronze or copper surfaces may be in contact with the dye-vat without injury to the color. Silk is frequently dyed in copper vats. The sensitiveness of most dyes to copper may be avoided by the placing of strips of zinc in the vat or by the addition of ammonium sulphocyanide. Yarn may also be dyed on machines, where the hanks are turned mechanically. Loose wool is frequently dyed by poleing in large round copper vats; it may also be dyed in rotating machines. Cloth is generally dyed in vats, being turned by means of a revolving winch; or it may be dyed on a jigger, consisting of two sets of rollers which roll the cloth on and off through the dye liquor. Warps are also dyed by machines. Yarn may also be dyed in cops by means of suitable machines so devised as to force the dye liquor through the cop by pressure or suction.

**5. Influence of the Water Employed in Dyeing.** — Water as employed in the dyehouse for the preparation of vats and the solution of the dyestuffs and various chemicals is generally obtained either from a river supply or from a well or spring. Rain-water is sometimes collected and employed for purposes where a very pure article is desired, such as for the solution of dyestuffs, etc.; it is usually not available in sufficient quantities nor regular enough in its supply to be serviceable for the preparation of dye-vats. Rain-water is considered as the purest form of natural water. Well and spring water are derived from rain-water which has passed through the surface of the earth until it has reached an impervious layer which causes it to collect in subterranean reservoirs from which it may be pumped as well-water, or it may flow underground until it eventually reappears at the surface as a spring. Such water usually contains various metallic salts in solution and generally has but little insoluble matter in suspension. The exact nature and amount of the dissolved substances will naturally vary considerably with the character of the soil and rock through which the water has

passed. Some rocks, like granite and gneiss, are very insoluble, and water percolating through these may be quite free from dissolved impurities, and springs or wells from such a source may be quite "soft." If the water, however, in its percolation through the soil passes through strata of limestone, chalk, sandstone, etc., some mineral compounds pass into solution, especially salts of magnesia and lime. Such a water is termed "hard." Iron compounds form a common constituent of soils and rocks, and consequently water that passes through such will be liable to contamination with iron; this will be more especially the case if at the same time it is in contact with decaying vegetable matter, as the latter furnishes certain organic acids which exert a strong solvent action on the iron compounds. Water containing a marked content of iron is termed "chalybeate" or "ferruginous."

River-water consists largely of surface-water, that is, rain-water which drains directly from the surface of the soil without percolating through the ground to any extent; besides this, river-water also contains well- or spring-water feeding into it from small streams, etc., having their origin in springs. The surface-water draining into a river is liable to bring into it a large amount of suspended matter, though not so much dissolved matter. The nature and extent of this suspended matter will, of course, vary largely with the season of the year and the character of the environments. From this it may be seen that river-water will, as a rule, contain more suspended matter and less dissolved matter than well-water. The suspended matter is comparatively easily removed, however, whereas the dissolved substances may give rise to considerable trouble.

The influence of the impurities in water on the dyeing operations will depend very largely on the character of the dyestuffs employed. Hard water containing lime and magnesia compounds, as a rule, does not interfere with the dyeing of colors in an acid bath, as the addition of the acid prevents any precipitation of the coloring-matter by the metallic salt. In certain cases the tone of the resulting color-lake may be somewhat modified by the presence of the mineral salts, but such is very rarely the



case. The presence of iron, however, even in very slight quantities, in the water, may cause a considerable alteration in the color, usually dulling and darkening it. With the general class of basic dyes hard water cannot be employed without suitable correction by the addition of acetic acid. The basic dyes form insoluble precipitates with lime and magnesia compounds which will result in a large loss of coloring-matter and also faulty and streaky dyeing by reason of the sticky precipitate of coloring-matter becoming smeared on the material being dyed. The presence of iron in the water is also very deleterious in using basic dyes. With the class of substantive colors the influence of hard water varies largely with the particular dyestuff, in some cases causing precipitation and in others not. As a general rule, however, it may be taken that hard water is deleterious with this class of dyes, and should be corrected by the addition of a suitable amount of soda ash in order to precipitate all the lime and magnesia compounds which may be in solution. The presence of iron is also bad, as it causes a discoloration of the dyestuff. With mordant dyes the use of hard water, if it does not contain any iron, is considered beneficial, as the lime present produces a better color-lake; in fact, unless the water is sufficiently hard, a soluble salt of lime is usually added in the dyeing of most alizarin colors. In certain cases where a dulled or "saddened" effect is desired, the presence of iron may be beneficial.

In mordanting operations, such as in the use of metallic salts on wool or silk, hard water may be used with impunity provided it does not contain any iron, which will result in the dulling of the eventual color-lake. In mordanting cotton with tannic acid, the use of hard water may be considered as somewhat beneficial, if anything, as it leads to a better fixation of the tannin mordant.

In bleaching operations on cotton, where chloride of lime or acids or caustic soda may be employed, the use of hard water is not injurious; though it should not be contaminated with iron. In the bleaching of wool with solutions of sodium bisulphite, hard water may also be employed. In all operations of scouring or bleaching where soap solutions are employed hard water should not be used, as the soap forms a highly insoluble and sticky precip-



itate with the mineral salts present in the water, causing thereby great loss of soap and the liability of serious faults in the textiles due to the precipitate of soap becoming incorporated with the fabrics. One part of lime present in hard water will precipitate about sixteen parts of ordinary soap.

When hard water is employed for the washing of fabrics, whether after scouring, dyeing, or bleaching, it may give rise to certain faults known as "lists" by reason of draining and evaporating unevenly of the hard water from the goods, thus leaving deposited in the material the dissolved mineral matter. Hard water, in fact, is probably more injurious in this connection than in most of the other operations of dyeing.

#### SAMPLES.

- 124. Naphthol Red EB on wool.
- 125. Emin Red on wool.
- 126. Tartrazine on wool.
- 127. Naphthol Yellow S on wool.
- 128. Alizarin Blue SAE on wool.
- 129. Acid Violet 5BF on wool.
- 130. Patent Blue V on wool.
- 131. Orange II on wool.
- 132. Wool Blue 2B on wool.
- 133. Acid Green on wool.
- 134. Brilliant Orange G on cotton.
- 135. Ponceau 4R on cotton.
- 136. Rose Bengale on cotton.
- 137. Methyl Blue on cotton.
- 138. Brilliant Croceine M on cotton.
- 139. Metanil Yellow on cotton.
- 140. Erythrosine B on cotton.
- 141. Irisamine G on cotton.
- 142. Acid Magenta on silk.
- 143. Acid Violet 4RS on silk.
- 144. Methyl Blue for silk on silk.
- 145. Orange II on silk.
- 146. Lyons Blue on silk.
- 147. Crystal Ponceau 6R on silk.
- 148. Cyanole BB on silk.
- 149. Acid Green on silk.
- 150. Azo Fuchsine on silk.
- 151. Silk Black 4BF on silk.

## QUIZ 8.

194. Name three red acid dyes, and compare the character of the colors they give.

195. What color does Tartrazine give on wool? Cyanole on silk? Brilliant Croceine is of what color?

196. What colors do Erythrosine and Irisamine give on cotton? What peculiarity is to be noticed in connection with these colors?

197. What do the letters after the names of dyestuffs represent? What would be the difference between Acid Violet 4R and Acid Violet 2R? Acid Green B and Acid Green G?

198. Name some of the more important dyestuff factories with the names of their agencies in the United States.

199. What agencies do the following abbreviations represent, and of what foreign factories are they the agents: Bad., Elb., Ber., Cass., Metz, Klp., RH., Bs.?

200. What precautions should be taken in the storage of dyestuffs in the mill?

201. How much actual dyestuff do paste dyes usually contain? What precautions should be taken in the preservation and use of these colors?

202. What is the method recommended in general for the dissolving of dyestuffs? Should the solid coloring-matter be added to the dye-bath directly? Explain why.

203. Can all dyestuffs be dissolved in boiling water without injury? Name some of these which cannot.

204. For difficultly soluble dyes how much water will be required for the solution of one part of dyestuff? How much for readily soluble dyes? About how much dyestuff can be dissolved in 10 gallons of water?

205. Why is it unsafe to use hard water in the dissolving of dyestuffs? What class of dyes are especially sensitive to hard water?

206. If hard water has to be employed for dissolving basic dyes, how should it be corrected? How are acid dyes dissolved in hard water?

207. How should hard water be treated for the solution of Water Blue, Alkali Blue, and Acid Violet? For the phthalein dyestuffs? For the substantive dyestuffs?

208. What two causes may lead to the precipitation of the dyestuff in standing baths?

209. How is yarn mostly handled in dyeing? What character of vat is employed? What metals only should be present in the dye-vat?

210. How may the sensitiveness of most dyes to copper be avoided, if it is necessary to have this metal in the vat?

211. Describe the general forms of machines employed for the dyeing of yarn. What advantages do machines possess over hand dyeing?

212. In what manner is loose wool or cotton dyed? Describe a form of machine for loose stock dyeing.

213. Describe the method employed for dyeing cloth. What is a "winch," a "jigger"?

214. What are "warps"? How is warp dyeing carried out?

215. What is meant by a "cop"? Describe the principle of a cop-dyeing machine. May other forms of material besides cops be dyed in similar machines?

216. If 75 kilos of wool are to be dyed with 0.08 per cent. of dyestuff, how many grams of coloring-matter would be required? If 100 pounds are to be dyed with the same percentage, how many ounces and grains of dyestuff would be necessary?

217. 250 grams per 100 kilos is how many ounces and grains per 100 pounds?

218. If 10 pounds of goods are to be dyed with  $\frac{1}{2}$  per cent. of dyestuff, how many grains of color would be necessary? What volume of dye solution containing 8 ounces of color per gallon would be required (in pints, gills, and noggins)?

219. Suppose 5-gram skeins of wool are dyed in tests using dye solutions containing 1 gram per litre; what percentage of color would 1 cc. represent? A color obtained with 20 cc. of the test solution would be equivalent to how many ounces and grains of dyestuff on 100 pounds of goods?

## SECTION IX.

### TESTING THE FASTNESS OF COLORS.

**Experiment 57. Fastness to Light.** — For this test (and those succeeding) use five of the skeins dyed in the preceding section. Cut off a sample of the dyed skein about three inches in length and place it in an exposure board, arranging it in such a manner that one-half of the sample is exposed to the light, while the other half is protected. Hang the exposure board on the inside of a window facing the south in order to obtain as much sunlight as possible. Allow the exposure to continue for one week, at the end of which time examine the sample for fading. If the color shows any perceptible alteration, it must be considered as *not fast* to light. If no fading is observed, the exposure should be continued for three weeks more. The sample is now examined a second time, and if any fading is apparent, the sample is removed and classified as *moderately fast*. If no fading is apparent the sample may be classified as *quite fast* to light (152, 153, 154, 155, 156). The degrees of fastness to light may also be classified numerically as follows:

1. Not faded in four weeks' exposure.
2. Not faded in one week's exposure.
3. Faded by one week's exposure.

**Experiment 58. Fastness to Washing.** — This is to represent the fastness of the color to scouring with soap. Take 5 or 6 strands of the dyed yarn to be tested and plait it with a similar amount of white woolen and white cotton strands, so as to make up a small test sample about 4 inches in length. This sample is then steeped in a soap solution containing 5 grams of soap per litre. About 50 cc. of the solution will be required for each test, and not more than one sample should be scoured in the same liquor. Have the temperature of the soap solution at 140° F.,



and wash the sample thoroughly by rubbing with the hands in the same manner as if the sample were dirty and you were trying to clean it. Use every precaution of cleanliness in order to prevent the sample from becoming stained with any other color. Then wash well in fresh warm water, and dry (157, 158, 159, 160, 161). Note if this treatment has caused the color to bleed into either the white wool or cotton with which the colored yarn is plaited, also if any of the color runs into the soap liquor, and after drying compare the sample with the original color and note if it has undergone any alteration.

**Experiment 59. Fastness to Fulling or Milling.** — Plait together a small test sample about 4 inches in length, using several strands of the dyed yarn and some strands of white woolen yarn. Work the sample so prepared in about 50 cc. of a solution containing 5 grams of soap and 2 grams of soda ash per litre at a temperature of 140° F. Rub the sample vigorously between the hands or between two pieces of wood in order to felt the fibres together and so imitate the action of fulling. After the fibres have been well felted together, wash the sample in fresh warm water, and dry (162, 163, 164, 165, 166). Note if the color has bled into the white wool or into the soap liquor; also compare the tested sample with the original color and note if it has undergone any alteration. The fulling test is only applicable to dyeings on wool.

**Experiment 60. Fastness to Water.** — This test is more especially applied to dyeings on silk and cotton yarns, and is to represent particularly fastness to rain. Plait together a small test sample about 4 inches in length, using strands of the dyed yarn with similar amounts of white silk and white cotton yarns. Steep this sample in distilled water for 12 hours (over-night), then squeeze out and dry (167, 168, 169, 170, 171). Note if the color has bled into either of the white yarns or into the steeping water.

**Experiment 61. Fastness to Perspiration.** — This is required of all clothing material intended for wear next to the skin; also for materials for making horse-blankets, etc. The action of perspiration is an acid one, and is said to be best represented chemically by the action of acetic or lactic acid on the color.

Plait a few strands of the dyed yarn with strands of white wool and cotton yarns in the usual manner, and steep for 1 hour in about 50 cc. of a solution containing 100 cc. of lactic acid (22 per cent.) per litre at the ordinary temperature. Then squeeze, wash and dry (172, 173, 174, 175, 176). Note if the color has bled into either of the white yarns or if the color has suffered any change from the original.

**Experiment 62. Fastness to Carbonizing.** — The carbonizing process is the treatment of woolen material with acid and then drying for the purpose of decomposing any vegetable matter present. It is especially used in connection with shoddy, though it is also at times employed on woolen piece-goods to remove specks of vegetable matter in the finished cloth. Take a small sample of the dyed woolen skein and steep it for 15 minutes in a solution of sulphuric acid at 4° Tw. and at a temperature of 140° F. Squeeze, and dry without washing, then wash well and dry again (177, 178, 179, 180, 181). Note if the color has undergone any alteration by this treatment. This test is only applicable to dyed woolen materials.

**Experiment 63. Fastness to Cross-Dyeing.** — By cross-dyeing is meant the dyeing of pieces containing white wool woven with dyed cotton yarn; the wool being dyed in a boiling acid bath, the dyed cotton must not be changed by the process. Make a small plaited sample of strands of the dyed cotton yarn with strands of white woolen yarn and boil the sample so prepared for 15 minutes in about 50 cc. of a solution containing 1 cc. of concentrated sulphuric acid and 2 grams of Glaubersalt per litre; wash well, and dry (182, 183, 184, 185, 186). Note if the color has undergone any alteration or if it has bled into the white yarn. This test is applied only to cotton dyeings.

**Experiment 64. Fastness to Stoving.** — Sometimes it is necessary to bleach woolen pieces containing white and colored yarns woven together in order to clear up the white yarns. This bleaching is done with sulphur dioxide (fumes from burning sulphur) as a rule, and the process is known as "stoving." Take a small sample of the dyed woolen yarn, moisten with water, and

hang it for 6 hours in a closed compartment containing sulphurous acid gas. Note if the color has undergone any alteration (187, 188, 189, 190, 191). This test is applied only to dyed woolen materials.

**Experiment 65. Fastness to Chloring.** — Cotton pieces containing white and dyed yarns woven together sometimes require the bleaching of the white after being woven. Towelling with colored borders is a good example of this class of material. The dyed colors must therefore stand a treatment with chloride of lime solution, and this is known as "chloring." Take a sample of the dyed cotton skein and steep for one-half hour in a cold solution of chloride of lime at  $\frac{1}{2}^{\circ}$  Tw., rinse off and pass through water slightly acidulated with sulphuric acid. Finally wash well and dry (192, 193, 194, 195, 196). Note if this treatment has caused any change in the color.

**Experiment 66. Fastness to Crocking or Rubbing.** — By this is meant that the dyed material should not stain white cloth when rubbed against it. Well-dyed material should seldom show this defect, although plush or pile fabrics sometimes rub considerably. If the washing of the material after dyeing has not been sufficiently thorough, so as to remove all particles of unfixed dyestuff, the color afterwards is liable to rub. This fastness is applicable to all classes of dyeing. Carry out the test by rubbing a dry sample of the dyed yarn vigorously on a piece of white calico and noting if it causes any smut on the white cloth (197, 198, 199, 200, 201).

The foregoing tests for fastness of dyed colors represent the principal requirements to be ordinarily met with. A careful study of the results of these tests will serve to show that fastness to any test, as well as the degree of that fastness, is not necessarily determined by the general class to which a dyestuff belongs, but is rather a particular property of the individual dyestuff. Furthermore, it is to be borne in mind that the requirements for fastness of colors are largely to be determined by the particular use to which the dyed material is to be put, and an intelligent discrimination in the selection of dyes to be used should be made with this in view. A more extensive discussion of the fastness of



dyed colors on various materials will be taken up in a succeeding section, after the various methods of dyeing have been more thoroughly studied.

*Tabulation of Results of Tests.*

Make records of the results of the various tests in the following manner, employing five different samples for the respective tests such as have been dyed in the previous section. The tested samples should be preserved in the student's sample book for future reference.

FASTNESS TO LIGHT.

No.	Dyestuff.	Fibre.	Fading observed in		Fastness.
			One week.	Four weeks.	
152	.....	.....			.....
153	.....	.....			.....
154	.....	.....			.....
155	.....	.....			.....
156	.....	.....			.....

FASTNESS TO WASHING.

No.	Dyestuff.	Fibre.	Stains.		
			Soap liquor.	White wool.	White cotton.
157	.....	.....			.....
158	.....	.....			.....
159	.....	.....			.....
160	.....	.....			.....
161	.....	.....			.....



## FASTNESS TO FULLING.

No.	Dyestuff.	Fibre.	Stains.		Change in color.
			Soap liquor.	White wool.	
162	.....	.....	.....	.....	.....
163	.....	.....	.....	.....	.....
164	.....	.....	.....	.....	.....
165	.....	.....	.....	.....	.....
166	.....	.....	.....	.....	.....

## FASTNESS TO WATER.

No.	Dyestuff.	Fibre.	Stains.		
			White cotton.	White silk.	Water.
167	.....	.....	.....	.....	.....
168	.....	.....	.....	.....	.....
169	.....	.....	.....	.....	.....
170	.....	.....	.....	.....	.....
171	.....	.....	.....	.....	.....

## FASTNESS TO PERSPIRATION.

No.	Dyestuff.	Fibre.	Stains.		Change in color.
			White wool.	White cotton.	
172	.....	.....	.....	.....	.....
173	.....	.....	.....	.....	.....
174	.....	.....	.....	.....	.....
175	.....	.....	.....	.....	.....
176	.....	.....	.....	.....	.....

## FASTNESS TO CARBONIZING.

No.	Dyestuff.	Alteration in color.
177	.....	.....
178	.....	.....
179	.....	.....
180	.....	.....
181 <sup>a</sup>	.....	.....

## FASTNESS TO CROSS-DYEING.

No.	Dyestuff.	Alteration in color.	Bleeding into white.
182	.....	.....	.....
183	.....	.....	.....
184	.....	.....	.....
185	.....	.....	.....
186	.....	.....	.....

## FASTNESS TO STOVING.

No.	Dyestuff.	Alteration in color.
187	.....	.....
188	.....	.....
189	.....	.....
190	.....	.....
191	.....	.....

## FASTNESS TO CHLORING.

No.	Dyestuff.	Alteration in color.
192	.....	.....
193	.....	.....
194	.....	.....
195	.....	.....
196	.....	.....

## FASTNESS TO CROCKING.

No.	Dyestuff.	Fibre.	Color rubbing on white.
197	.....	.....	.....
198	.....	.....	.....
199	.....	.....	.....
200	.....	.....	.....
201	.....	.....	.....

## SAMPLES.

- 152-156. Tests for fastness to light.  
 157-161. Tests for fastness to washing.  
 162-166. Tests for fastness to fulling.  
 167-171. Tests for fastness to water.  
 172-176. Tests for fastness to perspiration.  
 177-181. Tests for fastness to carbonizing.  
 182-186. Tests for fastness to cross-dyeing.  
 187-191. Tests for fastness to stoving.  
 192-196. Tests for fastness to chloring.  
 197-201. Tests for fastness to crocking.

## QUIZ 9.

220. Describe the method employed for the testing of the fastness of dyed colors to light. What standards of fastness are adopted?

221. Why should a southern exposure be given when testing samples for fastness to light? Why should the samples not be exposed to the outside atmosphere?

222. Is there any dyestuff of organic nature absolutely fast to light? Can you explain in any manner why light causes the fading of colors? Would colors fade under red light, and why?

223. What classes of goods require fastness to light more than others? Would fancy silks for ladies' wear require especial fastness to light, and why?

224. How would you test a dyed color in order to obtain its fastness to washing or scouring? What is the purpose of twisting white wool and cotton with the sample?

225. What is the strength of the soap solution employed for the scouring test, and at what temperature is the test conducted?

226. What character of dyeings require especial fastness to washing? Are dyeings on silk required to be particularly fast to washing?

227. Why must dyeings on loose wool or loose cotton be very fast to scouring? Explain why dyeings on weaving yarns should be fast to scouring.

228. What defects in the dyeing process may lead to poor fastness to washing of colors which would otherwise be fast?

229. What is meant by fulling or milling? Explain briefly how the process is carried out. Why has fulling a more severe action on the color than scouring?

230. Give the method for testing the fastness of a dyed color to fulling.

231. Are cotton and silk materials fulled? Explain why woollen materials felt together in fulling.

232. What classes of woollen materials are usually fulled? Why should dyeings on loose wool be fast to fulling, as a rule?

233. Is it often necessary that dyeings on worsted yarns should be particularly fast to fulling? Why should dyeings on woollen yarns be fast to fulling?

234. In dyeing piece-goods of woollen material is it necessary to select dyestuffs which are fast to fulling? What is meant by a level-dyeing dyestuff, and why is this class preferred for piece-goods?

235. Under what conditions may it be necessary to dye cotton yarns with colors fast to fulling? Are other solutions besides those containing soap and alkali ever employed for fulling?

236. What does fastness to water represent? How is the test carried out? To what materials is this especially applied?

237. If a color is fast to scouring, does it necessarily signify that it is also fast to water? Does fastness to fulling also imply fastness to scouring?

238. What classes of fabrics require colors having fastness to perspiration? What is the chemical action of perspiration?

239. Describe the method of testing a color for its fastness to perspiration. What kind of acid is lactic acid, and why is it used in the perspiration test?

240. What is meant by "carbonizing" as applied to textiles? Briefly outline the method of carbonizing.



241. What classes of materials are carbonized? Describe the method employed in testing the fastness of a color carbonizing.
242. Would colors on cotton materials ever require to be fast to carbonizing? What other processes besides treatment with solutions of sulphuric acid are employed for carbonizing?
243. What is meant by "cross-dyeing"? How is the test representing fastness to cross-dyeing carried out?
244. The dyeings on what fibres are tested for fastness to cross-dyeing? How does the test for cross-dyeing differ from that of the carbonizing test?
245. What is meant by stoving? How is the process conducted? What classes of materials are subjected to stoving?
246. Describe the method of testing a color for fastness to stoving. What is the chemical action of sulphurous acid on coloring-matters?
247. What is meant by chloring? What kinds of dyed materials are sometimes chlored? What is the object of chloring?
248. How is the test for bleaching or chloring conducted? Why is it necessary to treat with acidulated water?
249. What is meant by "crocking"? How is this test conducted? Should well-dyed materials crock?
250. What classes of dyed materials are liable to crock, and explain why? What faults in dyeing may lead to lack of fastness of the color to crocking?
251. What qualities of fastness should dyeings on loose wool possess? Dyeings on shoddy?
252. What should be the requirements for fastness of worsted weaving yarns for trouserings? Of worsted knitting yarns? Of hosiery yarns in general?
253. What requirements would be expected in colors dyed on yarns intended for flannels, blankets, carriage and steamer rugs, and plaid dress-goods?
254. Yarns employed for carpets and tapestries require colors possessing what qualities of fastness? Yarns for plushes and velvets require what fastness?
255. Fancy weaving yarns of cotton for all cotton dress-goods and shirtings require what qualities of fastness? Dyed cotton yarns to be employed in union goods should be fast to what tests?
256. Dyes for bookbinder's cloth should have what qualities of fastness? Dyed cotton yarns woven in towelling should be fast to what tests?

## SECTION X.

### APPLICATION OF BASIC DYES TO WOOL AND SILK.

**Experiment 67. General Method of Applying Basic Dyes to Wool.** — Basic colors are usually dyed on wool in neutral or slightly acid baths. Dye a skein of woollen yarn in a bath containing 300 cc. of water, 2 per cent. of acetic acid, 10 per cent. of Glaubersalt, and 1 per cent. of Methylene Blue; enter at 100° F., gradually raise to 180° F., and dye at that temperature for one-half hour (202). Dye another skein of woollen yarn in a similar bath containing 10 per cent. of Glaubersalt and 1 per cent. of Methylene Blue in the same manner, and note that the dyestuff is absorbed more rapidly (203). The function of the acid is to retard the dyeing, and so assist in the even distribution and thorough penetration of the color. If the bath in the first case does not exhaust completely, lift the skein and add 4 per cent. of borax, and continue dyeing for 15 minutes. The borax is a mild alkali, and is added for the purpose of neutralizing the acid in the bath and so permitting the complete exhaustion of the dyestuff. Acetic acid is better to use in dyeing basic colors than sulphuric acid, as the former is volatile, and as the temperature of the dye-bath rises the acidity becomes lessened and consequently the exhaustion is better.

**Experiment 68. Showing the Effect of Hard Water on Basic Dyes.** — Dye a skein of woollen yarn in a bath containing 300 cc. of water and 1 per cent. of Magenta; enter at 100° F., gradually bring to 180° F., and dye at that temperature for one-half hour (204). At the same time dye a second skein in a similar bath to which, however, there is added 5 cc. of lime water (205). Carry out the dyeing in the same manner, and note any difference in the appearance of the two skeins after dyeing. Lime salts in water

cause a precipitation of basic dyes in the bath, and hence interfere with their dyeing properties.

**Experiment 69. Showing the Greater Coloring Power of Basic Dyes over Acid Dyes.** — Dye a skein of woollen yarn in a bath containing 300 cc. of water, 10 per cent. of Glaubersalt, 4 per cent. of sulphuric acid, and 2 per cent. of Acid Magenta; enter at 140° F., gradually raise to the boil and dye at that temperature for one-half hour (206). Dye a second skein in a bath containing 300 cc. of water, 10 per cent. of Glaubersalt, and 2 per cent. of Magenta; dye for the same length of time and under similar conditions as above (207). After dyeing, wash and dry the two skeins. Note the depth of color on each, and it will be found that the skein dyed with the acid color is considerably lighter than the one dyed with the basic color.

**Experiment 70. Use of a Neutral Bath.** — Most basic dyes will dye fairly well on wool from neutral baths, though the water used should be soft, or, if hard, should be corrected by the addition of acetic acid. For each degree of hardness of the water about  $\frac{3}{4}$  oz. of acetic acid should be added per 100 gallons of water. Or perhaps a more convenient method is to add acetic acid to the water of the dye-bath until it shows a faint acid reaction with litmus paper (turning blue litmus paper red). The color is more apt to be uneven from neutral baths than from those containing acid. Dye a skein of woollen yarn in a bath containing 300 cc. of water, 10 per cent. of Glaubersalt, and 1 per cent. of Rhodamine; enter at 100° F., gradually bring to 180° F., and continue at that temperature for one-half hour. Wash and dry (208).

**Experiment 71. Dyeing Silk with Basic Colors.** — Dye a test-skein of silk in a bath containing 150 cc. of water, 25 cc. of boiled-off liquor, and 2 per cent. of Magenta; enter the skein at 120° F., and gradually bring to 180° F., and dye at that temperature for one-half hour, then wash well and dry (209). Silk, like wool, has a direct affinity for the basic colors.

**Experiment 72. Dyeing Silk in a Neutral Soap Bath.** — Prepare a bath containing 150 cc. of water, 5 per cent. of soap (which



should be a neutral, olive oil soap). Work the test-skein of silk in this bath for a short time at  $140^{\circ}$  F., then add 2 per cent. of Methylene Blue solution in several portions, at the same time raising the temperature of the bath to  $180^{\circ}$  F. Continue at this temperature for 15 minutes, then add sufficient acetic acid to slightly acidulate the bath, and continue dyeing for 15 minutes longer. Then wash well and brighten with acetic acid in the manner given in Exp. 51 (210). Dye a second sample of silk in the same manner with 2 per cent. of Rhodamine (211).

**Experiment 73. After-Treatment of Basic Dyes on Silk with Tannin.** — Dye a test-skein of silk with 2 per cent. of Methyl Violet as described in Exp. 72; wash well (212) and pass into a fresh bath containing 100 cc. of water and 1 gram of tannic acid; work for 20 minutes at  $180^{\circ}$  F., then sink under the liquor and leave for one-half hour without further heating. Squeeze out the excess of liquor and work in a fresh bath containing 100 cc. of water and 0.5 gram tartar emetic at a temperature of  $140^{\circ}$  F. for 20 minutes. Wash well and brighten with acetic acid as usual (213). Dye a second skein of silk in the same manner with 2 per cent. of Malachite Green (214).

#### NOTES.

1. **The Use of Basic Dyes on Wool and Silk.** — The basic dyes are very largely employed on silk, for the silk fibre has a strong affinity for these coloring-matters, and the methods of their application are simple. Furthermore, the colors obtained with the basic dyes are especially characterized by depth and brilliancy, factors which are very important in dyeing silk. On account of the strong affinity of basic colors for silk, it is not always wise to add all of the required dyestuff at once to the bath, but in several portions as the dyeing proceeds. Especial care must be taken in the solution of the basic dyes, as if boiling water is employed for this purpose there is liability of tarry matters being formed by a partial decomposition of the dyestuff. It is best to use warm water acidified with acetic acid. In some cases the dyes are more readily dissolved if a little methylated alcohol is added. Acetin,



which is a preparation from acetic acid and glycerin, is sometimes employed for assisting the solution of basic dyes. The basic dyes at the present time do not find much application on woolen goods, as better results are generally to be obtained by the use of acid colors. In former years, the basic colors were much more used than they are now for wool; but by treatment with sulphuric acid many of the basic dyes can be converted into corresponding acid derivatives which are used instead. Thus, Magenta gives Acid Magenta, Malachite Green furnishes Acid Green, Methyl Violet is converted into Acid Violet, etc. The affinity of basic dyes for wool is very great, hence they are liable to dye up uneven unless proper precautions are taken, such as adding the dye solution to the bath in several portions, starting the dyeing at a low temperature, adding some acetic acid or alum to the bath, etc. For the same reason the basic dyes give good exhaustion in the dye-bath, and it is seldom necessary to employ standing baths. The temperature when dyeing basic colors is seldom brought to over 180° F., as higher temperatures may cause decomposition of the dyestuff (especially noticeable in the case of Auramine), leading to the formation of sticky, insoluble, tarry products. As the basic colors are quite sensitive to hard water, it is always necessary to correct such water by the proper addition of acetic acid. Rhodamine is quite extensively used on wool for the production of bright pink colors; the dyed material may be bleached with sulphurous acid gas, which considerably brightens up the color and also gives it greater fastness to light. Rhodamine pinks are sufficiently fast to washing for most purposes. The basic colors are especially serviceable in the dyeing of weighted silks where the weighting has been done with tin salts. The after-treatment with tannic acid and tartar emetic is for the purpose of giving greater fastness to washing. Colors obtained with basic dyes on wool are as a rule not very fast to light or washing, and also exhibit a tendency to crock. Dye spots, consisting of uneven streaks or spots, often occur when dyeing with basic colors, caused by precipitation of the color-base in the dye-bath either by the use of hard water or by imperfect solution.

**SAMPLES.**

- 202. Showing application of basic dye on wool.
- 203. Dyed with glaubersalt in bath.
- 204. Magenta dyed with soft water.
- 205. Magenta dyed with hard water.
- 206. Dyeing with 2 per cent. Acid Magenta.
- 207. Dyeing with 2 per cent. Magenta (basic).
- 208. Rhodamine dyed in neutral bath.
- 209. Showing application of basic dye to silk.
- 210. Dyeing silk in a neutral soap bath.
- 211. Rhodamine on silk.
- 212. Methyl Violet before treatment with tannin.
- 213. Methyl Violet after treatment with tannin.
- 214. Malachite Green on silk.

**QUIZ 10.**

257. In what character of dye-bath are basic dyes usually applied to wool? How does the application of basic dyes differ from that of acid dyes?
258. At what temperatures are basic colors generally dyed? Explain the reason for this.
259. When acid is added to the dye-bath in dyeing basic colors, what is its function? What acid is best to employ, and why?
260. Why is borax at times added in dyeing basic colors? Borax has what chemical character? What is borax?
261. What is meant by "hard" water? What action does hard water have on basic colors?
262. Of acid and basic dyes, which have the greater coloring power? Compare Acid Magenta and Magenta for intensity of coloring power.
263. How may hard water be corrected for use in dyeing basic colors? What is meant by a "degree" of hardness?
264. How much acetic acid per 100 gallons of water is necessary to add to correct each degree of hardness? If the degrees of hardness of the water are not known, how could you tell how much acetic acid to add to the water?
265. What color does Rhodamine give on wool, and how is it dyed? What peculiarity is to be noticed with respect to this color?
266. What is the general method for dyeing silk with basic colors? What is boiled-off liquor? What is its function in the dye-bath? What substitutes may be employed in its place?
267. Give the method for dyeing silk in a neutral soap bath. What kind of soap should be used? Why is acetic acid added at the end of the dyeing operation?

268. What is meant by "brightening" the silk after dyeing, and how is this done?

269. How may basic dyes on silk be after-treated? Give the details of the process. What is the purpose of the after-treatment?

270. Are basic dyes at the present time largely employed on wool and silk? What are the special characteristics of the colors obtained by the use of basic dyes?

271. What precautions should be taken to obtain level shades on silk with the basic dyes? Why are they liable to dye up unevenly?

272. What precautions should be taken in dissolving basic dyes? Should boiling water be used, and why?

273. Why is acetic acid added to the water in dissolving basic dyes? If the dyes are but slightly soluble in water what solvent should be used?

274. What is meant by "methyiated spirits"? What is "acetin," and of what use is it in connection with basic dyes?

275. Why are basic dyes not much employed in wool dyeing at present? How may basic dyes be converted into acid dyes? Mention some examples.

276. Why are basic colors liable to come up uneven on wool, and how may this be prevented? Do basic dyes give good exhaustion?

277. Why are most basic dyes not dyed at the boil? Why is alum sometimes added to the dye-bath?

278. How may a clear bright pink color be obtained on wool? Why is the dyed material afterwards stoved or sulphured, and what effect has this on the fastness of the color?

279. In what class of silk dyeing are the basic colors especially serviceable, and why? How may greater fastness to washing be obtained?

280. What disadvantages do basic dyes, as a class, have in wool dyeing? What is meant by dye spots; what causes them, and how may they be prevented?



## SECTION XI.

### BASIC DYES ON COTTON.

**Experiment 74. General Method of Dyeing.** — As cotton does not possess acidic properties, it does not combine directly with basic dyes, but requires an acid substance (mordant) to be added to the fibre in order for the dyeing to take place. Cotton readily absorbs tannic acid from solution, and as this acid forms good color-lakes with the basic dyes, it is a very suitable mordant for cotton in this connection. To illustrate this reaction, proceed as follows: Steep a skein of cotton yarn in a bath containing 300 cc. of water and 2 per cent. of tannic acid; enter at  $120^{\circ}$  F., raise to  $190^{\circ}$  F., then allow the skein to remain immersed in the bath without further heating, as it is found that the maximum amount of tannic acid is absorbed from a cooling bath. Now squeeze the skein (215), and together with an unmordanted skein of cotton yarn pass into a dye-bath containing 300 cc. of water and 1 per cent. of Methylene Blue; enter at  $100^{\circ}$  F., gradually raise to  $190^{\circ}$  F., and dye at that temperature for one-half hour; wash well and dry. It will be found that the mordanted skein (216) has become dyed, whereas the other skein (217) has only become slightly tinted. As tannic acid is liable to suffer decomposition at the boil, giving rise to brown coloring-matters and resinous products, it is not recommended to boil the mordanting bath, as the shade eventually obtained will probably be dulled. The tannin, by this method of treatment, is not held in an insoluble state in the cotton, so that when the goods are placed in the dye-bath some of the tannic acid passes again into solution in the dye liquor, causing some of the dyestuff to be precipitated and also causing a loss of color to the fibre. Hence it is customary to fix the tannic acid in an insoluble condition on the fibre before passing into the dye-bath, as will be described in a succeeding



experiment. Tannin is a vegetable astringent principle and occurs in many plants or vegetable extracts, such as sumac (containing about 20 per cent. of tannic acid), cutch (containing about 40 per cent. of tannic acid), etc. These vegetable extracts may be used in place of tannic acid itself, provided sufficient amount of them be taken to give the proper amount of actual tannic acid. Many of these vegetable extracts, however, also contain more or less brown coloring-matters associated with the tannin, and these are absorbed by the cotton, causing the latter to become considerably colored in the mordanting.

**Experiment 75. Fixing Tannin on Cotton with Tartar Emetic.—**

In order to fix the tannin mordant absorbed by the cotton from the mordanting bath so that it will not dissolve into the dye-bath, it is best to combine it with some metallic base and so form an insoluble tannate. Most of the tannates of the metals are dark in color, hence unsuitable for dyeing, except for the production of a limited range of shades. The tannate of antimony, however, possesses but very little color, and scarcely affects the resulting color of the dye. Tartar emetic is potassium antimony tartrate, and it is the antimony oxide which is present in the salt which serves the purpose of fixing the tannin; that is, the tannin reacts with the tartar emetic to form antimony tannate. Proceed as follows: Mordant a skein of cotton yarn in a bath containing 300 cc. of water and 2 per cent. of tannin as before described; squeeze and pass into a fresh bath containing 300 cc. of water and 1 per cent. of tartar emetic; work cold for 15 minutes. Then wash well in fresh water (218) to remove any excess of the antimony compound and any unfixed tannin, and pass to a dye-bath containing 300 cc. of water, 1 per cent. of Methyl Violet, and 2 per cent. of acetic acid; enter at 100° F., gradually raise to 190° F., and dye at that temperature for one-half hour (219). The amount of tannin used in mordanting should be about twice that of the dyestuff, and the amount of tartar emetic should be about one-half that of the tannin. The acetic acid is employed for the purpose of retarding the dyeing, so as to promote even and well penetrated colors.

**Experiment 76. Fixing Tannin with Copperas.** — Copperas is iron sulphate, and as it occurs in the form of green crystals, it is known as "green vitriol." Salts of iron combine with tannic acid to give black tannate of iron, hence tannin fixed on cotton with copperas or other iron salts gives the fibre a gray to black color, which, of course, affects the shade eventually dyed on the mordant. Mordant a skein of cotton yarn in the manner described above with 2 per cent. of tannin, squeeze, and steep for 15 minutes in a cold bath containing 300 cc. of water, 5 per cent. of copperas and 5 per cent. of whiting. The latter is calcium carbonate or chalk, and is added in order to keep the bath neutral, for when the tannic acid combines with the iron of the copperas there is liberated some sulphuric acid, and as tannate of iron is soluble in sulphuric acid, it will be redissolved. The chalk in the bath combines with the sulphuric acid as fast as formed, and thus keeps the bath neutral, so that the iron is able to combine fully with the tannic acid. Wash the mordanted skein, which will now have a gray or slate color, and preserve a sample for comparison (220), then dye the rest of the skein in a bath containing 300 cc. of water, 1 per cent. of Methylene Blue and 2 per cent. of acetic acid in the usual manner. Wash and dry (221). In the same bath with this skein also dye a skein of cotton yarn which has been mordanted in the usual manner with tannin and fixed with tartar emetic. Notice the difference in the colors obtained, due to the iron mordant; also compare the mordant color with the dyed color, and note the influence of the bottom color of the mordant on the resulting color-lake.

**Experiment 77. Use of Other Agents in Dyeing Basic Dyes.** — Mordant a test-skein of cotton yarn in a bath containing 300 cc. of water and 20 per cent. of sumac extract. Enter at 190° F., work the cotton in the bath for 15 minutes, then steep under the liquor for 1 hour without further heating. Squeeze the skein and pass into a fresh bath containing 300 cc. of water and 2 per cent. of antimony salt (a double salt of antimony fluoride with ammonium sulphate); work cold for 15 minutes, then wash well (222) and dye in a fresh bath containing 300 cc. of water,

5 per cent. of alum, and 2 per cent. of Thioflavine T. Conduct the dyeing operation as usual. Wash well and dry (223).

**Experiment 78. Dyeing Basic Colors in One Bath.** — Prepare a cold bath containing 300 cc. of water, 6 per cent. of acetic acid, 2 per cent. of tannic acid, and 1 per cent. of Malachite Green. Dye a skein of cotton yarn in this bath cold for 15 minutes, then raise the temperature to 105° F. for 15 minutes, and finally to 140° F. for 15 minutes; then rinse the skein, squeeze, and dry (224). The fastness to washing of the colors dyed in this manner may be increased by first rinsing after dyeing in water containing  $\frac{1}{2}$  to 2 per cent. of tartar emetic. This method is only applicable to amounts of dyestuff up to about 1 per cent. The color-lake is held in solution by the presence of the acetic acid, and only separates out gradually in the fibre on heating the bath.

**Experiment 79. Use of the Janus Dyes.** — These dyestuffs are basic colors which also possess substantive or direct dyeing properties, though to form a fast color-lake it is necessary to fix the dye with tannin. Prepare a dye-bath containing 300 cc. of water, 2 per cent. of acetic acid, 5 per cent. of zinc sulphate, and 2 per cent. of Janus Red. Add only a portion of the dyestuff solution at first; enter the cotton skein at about 200° F., work for 10 minutes, then add the remainder of the dyestuff; work for 10 minutes longer, and then add 20 per cent. of common-salt, and work for one-half hour at the boil. Rinse the dyed cotton (225), and pass into a fixing bath containing 300 cc. of water, 4 per cent. of tannic acid; work cold for 15 minutes; then lift the skein and add to the bath 2 per cent. of tartar emetic and 1 per cent. of sulphuric acid, and work cold for 15 minutes longer, then raise the temperature to 140° F. for 15 minutes. Finally wash well and dry (226).

#### NOTES.

**Substances Employed for Mordanting Cotton.** — *Tannins.* By the general term "tannins" is meant a number of related organic acids which occur as the astringent principles in vegetable life. They are generally analogous in their chemical properties and are characterized by their property of tanning animal skins (that is,



converting the animal tissue into leather), forming insoluble compounds with albumen, precipitating basic dyes from solution, and yielding bluish or greenish black colors with solutions of iron salts. The majority of the natural tannins also contain yellowish or brownish coloring-matters; pure tannic acid, however, has no special color. Some of the tannins, such as decoctions of gall-nuts and extracts of sumac, may be almost entirely decolorized by proper methods of treatment. Where delicate and bright colors are to be obtained on cotton with basic dyes it will be necessary to employ either pure tannic acid or a decolorized sumac extract.

Though cotton is in general very inert towards solutions of organic acids, it appears to possess considerable affinity for tannic acid, and will absorb it readily from its solutions. Tannins should be stored in a dry place, as continued exposure to damp air will cause the tannic acid to decompose, giving brownish-colored resinous substances. The following are the most important tannins employed in the mordanting of cotton: (1) *Tannic acid*, or gallo-tannic acid, is prepared especially from gall-nuts, which are very rich in this acid. Tannic acid comes on the market in the form of a light brown powder or yellowish to brownish scales which usually darken somewhat on exposure to light. It is soluble in 6 parts of cold water, and in even a less quantity of hot water; it is also freely soluble in alcohol, dilute acetic acid, and glycerin. Solutions of tannic acid, and also of any of the tannins, will gradually undergo fermentation and become destroyed. In order to prevent this decomposition in standing baths used for mordanting, it is advisable to boil up the baths repeatedly or to add a small amount of carbolic acid to them. When used as a standing bath about 70 per cent. of the amount of tannin originally added to the first bath should be used for replenishing. (2) *Sumac* is next in importance to tannic acid itself for purposes of dyeing cotton. The sumac from the *Rhus coriaria* is considered the best and it contains gallo-tannic acid. Sicilian sumac is the best and least colored variety; after this comes the American (Virginian) sumac, which can now be



obtained in very good qualities. Commercial sumac usually consists of the whole or the crushed or pulverized leaves, though the stalks and small stems are frequently admixed. Good qualities have an olive-green color and a pleasant smell; they contain from 15 to 20 per cent., and sometimes as high as 25 per cent. of tannin. Sumacs which are dull in color and of a musty smell have deteriorated by exposure to moist air and prolonged storing. Sumac contains a small amount of dull reddish brown coloring-matter, which prohibits its use in most cases for light and brilliant shades, so that it is chiefly employed for dark shades. Sumac extract is a thick dark brown liquid or paste, usually of about 52° Tw. density. It also occurs in the solid state. Decolorized sumac extracts are also to be had, and may be used in place of pure tannic acid for light colors. Liquid sumac extracts are very liable to fermentation, especially if kept in a warm moist room.

(3) *Galls*, or gall-nuts, are ball-shaped excrescences which grow on various plants, especially oak trees, and result from the sting of an insect in depositing its eggs. Of the oak-galls, the green or black Aleppo galls and the Turkish or Levant galls are the best and contain about 55 to 60 per cent. of gallo-tannic acid. Chinese and Japanese galls contain up to 80 per cent. of gallo-tannic acid, and these are principally used for the production of pure tannic acid.

(4) *Myrobolans* consist of the fruit of several Chinese and Indian plants, and they occur in trade in the dry state; they contain 25 to 45 per cent. of tannin and a yellowish brown coloring matter. They are not much used in this country, though sometimes employed for dyeing cotton black.

(5) *Divi-divi* is the fruit of certain plants in Central and South America; they contain 20 to 35 per cent. of tannin, and are used in the same way as myrobolans. There are many other tannin substances which are more or less locally employed where they are to be obtained in abundance, but the above-mentioned varieties are the principal ones to be met with in trade. In the mordanting of cotton for dyeing, 1 pound of pure tannic acid is equivalent to about 1½ to 2 pounds of gall-nuts, or 4 pounds of sumac extract of 25 per cent. strength, or to 5 to 6 pounds of sumac leaves.

*Tartar Emetic and Antimony Salts.* — Tartar emetic is the double tartrate of antimony and potassium; it is a crystalline salt and is not very soluble in cold water, but it is rather easily soluble in hot water. One part of the salt requires about 13 parts of water for solution at 70° F. and only about 3 parts of water at 180° F. The active principle in tartar emetic which enters into the fixation of the tannin in the mordanting of cotton is the antimony trioxide,  $\text{Sb}_2\text{O}_3$ , of which the pure salt contains 43.4 per cent. The commercial product consists of fine crystals of irregular lumps containing about 43 per cent. of antimony trioxide. It is frequently adulterated with cheaper substances. Though tartar emetic and the rest of the salts of antimony are poisonous, no ill effects need be feared from its use in dyeing, if the goods are well washed after mordanting. As tartar emetic is rather an expensive chemical, it is often replaced by cheaper salts of antimony, which have the same effect in the fixation of the tannic acid. The chief substitutes are as follows: (1) *Antimony salt*, which is the double salt of antimony fluoride and ammonium sulphate; it occurs as white crystals, of which 140 parts are soluble in 100 parts of water. The solution is strongly acid and corrodes glass and metals, owing to the hydrofluoric acid liberated. Antimony salt contains 47 per cent. of antimony trioxide; hence 9 parts are equivalent to 10 parts of tartar emetic. (2) *Patent salt*, or *Double antimony fluoride*, is antimony-sodium fluoride. It is also crystalline and readily soluble, and likewise corrodes glass and metals. It contains 66 per cent. of antimony trioxide; hence 65.8 parts of this salt are equivalent to 100 parts of tartar emetic. Of these two double fluorides of antimony, 5 to 20 parts are dissolved in 1000 parts of water, and their strong acidity is neutralized by the addition of 6 to 8 per cent. in weight of soda ash, or just enough to render the bath slightly turbid. (3) *Antimony oxalate* is the double oxalate of potassium and antimony, and was introduced as the first cheap substitute of tartar emetic; it has given much satisfaction, but has been nearly superseded by the double fluorides. It occurs as crystals which are readily soluble in water, but which dissociate rapidly into an insoluble

basic oxalate of antimony and a soluble binoxalate. It contains only 25.1 per cent. of antimony trioxide, as against 43.4 per cent. in tartar emetic; though it is claimed to replace equal weights of the latter, as it combines more rapidly with tannic acid. (4) *Antimonine* is the double lactate of antimony and calcium. It occurs as crystals containing 15 per cent. of antimony trioxide; it is hygroscopic and very readily soluble. It should be employed in a weakly acid solution, that is, with the addition of about 2 gallons of acetic acid per 1000 gallons of liquor. This product is quite extensively employed.

The fixing bath of tartar emetic, like that of the tannin, may be employed continuously, being freshened up accordingly. As the bath becomes acid on using, due to the removal of the antimony trioxide, a little soda ash should be added from time to time to neutralize the acid as it accumulates; this is best done by adding a dilute solution of soda ash until a slight turbidity is apparent. If the liberated tartaric acid is allowed to accumulate without being neutralized, it will act so as to redissolve the precipitated antimony tannate, and thus lessen the value of the fixing bath.

### SAMPLES.

- 215. Cotton mordanted with tannic acid.
- 216. Mordanted cotton dyed with Methylene Blue.
- 217. Unmordanted cotton dyed with Methylene Blue.
- 218. Cotton mordanted with tannin and antimony.
- 219. Methyl Violet on tannin-antimony mordant.
- 220. Cotton mordanted with tannin and copperas.
- 221. Methylene Blue on tannin-iron mordant.
- 222. Cotton mordanted with tannin and antimony salt.
- 223. Thioflavine T on tannin-antimony mordant.
- 224. Malachite Green dyed in one bath.
- 225. Janus Red before mordanting.
- 226. Janus Red after mordanting with tannin-antimony.

### QUIZ II.

281. In order that cotton may combine with basic dyes how must it be treated? The mordants must possess what chemical character?

282. Why is tannic acid a suitable mordant for cotton in connection with basic dyes? What other mordants could be used on cotton for basic dyes?



283. Describe the method of mordanting cotton with tannic acid. Why is the cotton allowed to cool down in the bath?

284. Does the cotton exhaust the tannic acid from solution? How much does it take up?

285. Give the conditions of dyeing the basic colors on cotton mordanted with tannic acid. At what temperatures does the dyeing take place?

286. Why is it recommended not to boil the tannin bath in mordanting cotton? Does the fibre hold the tannin in an insoluble condition after mordanting?

287. What is tannic acid and from what is it obtained? What other substances besides tannic acid do most vegetable tannins contain?

288. What is the object of fixing the tannin after mordanting and before dyeing? The salts of what metals are most suitable for fixing agents for tannin, and why?

289. What is tartar emetic? What is the active substance in this salt which acts as the fixing agent for the tannin?

290. What compound is formed in the fibre when tannic acid is fixed with tartar emetic?

291. Describe the method of fixing tannin on cotton with tartar emetic. At what temperature is the bath used? Why does a separate bath from the mordanting have to be used?

292. Explain the chemical reaction which takes place between the tartar emetic and the tannic acid. What color is the cotton after fixing with anti-mony?

293. Why is acetic acid added to the dye-bath in dyeing cotton with basic colors? What other substances may be employed for the same purpose?

294. Why should the cotton be thoroughly washed after the tannin mordant has been fixed with tartar emetic? Should it be washed after mordanting with tannin and before fixing?

295. How much tannin and tartar emetic should be used for mordanting cotton for each percentage of dyestuff used?

296. Suppose 50 pounds of cotton yarn are to be dyed with  $2\frac{1}{2}$  per cent. of Methyl Violet: give the size of vat required, if it is to be 3 feet deep by 2 feet wide. How much tannic acid and how much tartar emetic would be required? If sumac were used in place of tannic acid, how much would be required? How many quarts of acetic acid (sp. gr. 1.031) of 30 per cent. strength would be used? How many quarts of Methyl Violet solution containing 4 ounces per gallon?

297. What is copperas? By what other name is it commonly known in trade?

298. What reaction takes place between tannic acid and salts of iron? What color is the resulting compound?



299. Give the method of mordanting cotton with tannin and copperas. What class of shades may be dyed on this mordant?

300. What is whiting, and why is it employed in the fixing bath with copperas? Explain the chemical reactions which take place.

301. What color does Methylene Blue give on the tannin-iron mordant? How does the color compare with that obtained with the same dyestuff on a tannin-antimony mordant?

302. How much sumac extract is necessary for the mordanting of cotton? How does the color of the sumac mordant compare with that of the pure tannic acid mordant?

303. What color does Thioflavine T give on mordanted cotton? What is the purpose of the addition of alum to the bath?

304. Give the method of dyeing basic colors in one bath. What is the maximum amount of dyestuff which may be used in this method? What is the function of the acetic acid in the bath? How may the colors be made faster to washing?

305. What properties does the class of Janus dyes possess? How are these colors dyed on cotton? Why is the dyestuff to be added in several portions? How is the after-mordanting process for the Janus colors carried out?

306. What is meant by the general term "tannins"? What are their general chemical reactions?

307. What kind of coloring-matters do most natural tannins contain? Can vegetable tannin extracts be decolorized, and how?

308. When delicate or bright shades are to be obtained on cotton with basic dyes, what character of tannin must be used in the mordanting?

309. What precautions should be taken in the storage of tannins? What is the action of damp air on tannin extracts?

310. Name the principal tannin materials employed in the mordanting of cotton.

311. From what is tannic acid itself chiefly prepared? How does it occur in trade? Give some idea as to its solubility in water. In what other reagents is it soluble?

312. Are solutions of tannic acid at all stable? How should standing baths of tannic acid be preserved? How much tannic acid does cotton absorb from the first bath, and consequently what addition of tannin is needed for successive standing baths?

313. From what plant is sumac obtained? Which variety of sumac is considered the best? How does American sumac compare with others?

314. Of what does commercial sumac usually consist? What should be its appearance? What percentage of tannic acid does it contain?

315. Does sumac contain any coloring-matter, and can it be used for light and brilliant shades?

316. How does sumac extract occur in trade? Are sumac extracts liable to deterioration on storage?

317. What are galls or gall-nuts, and how do they originate? How much tannin do the best galls contain?

318. Of what do myrobolans consist? How much tannin do they contain? For what kind of dyeing are they sometimes used?

319. What is divi-divi, and how much tannin does it contain? Give the ratio in tannin between pure tannic acid, gall-nuts, sumac extract, and sumac leaves.

320. How does tartar emetic occur in trade? Give some idea as to its solubility in water. How much antimony trioxide should pure tartar emetic contain? How much is usually present in the commercial variety?

321. Are salts of antimony poisonous? Do they appear to give any bad results if properly employed in dyeing? Name the chief substitutes of tartar emetic.

322. Of what does antimony salt consist? How does it compare with tartar emetic as to solubility and the amount of antimony trioxide it contains? Of what character is its solution?

323. What is "patent salt"? How does it compare with tartar emetic as to solubility and amount of antimony trioxide present? How should the solutions of the antimony fluorides be prepared?

324. Of what does antimony oxalate consist? How does it react when dissolved in water? How does it compare with the antimony fluorides and tartar emetic?

325. What is antimonine? How does it compare with tartar emetic as to solubility and percentage of antimony trioxide present? How should its solution be prepared?

326. May the fixing baths of antimony salts be employed as a standing kettle, and if so, how should the baths be treated in order to prevent their deterioration?

## SECTION XII.

### REPRESENTATIVE BASIC DYES.

**Experiment 80. Principal Basic Dyes on Cotton.** — Use test-skeins of cotton yarn mordanted in the manner described in Exp. 75 with 4 per cent. of tannin and 2 per cent. of tartar emetic. Prepare the dye-bath with 2 per cent. of alum and the dyestuffs given below; enter at 100° F., gradually raise the temperature to 180° F., and dye at that point for one-half hour; wash well and dry.

- 2 per cent. Bismarck Brown (227).
- 2 per cent. Safranine (228).
- 2 per cent. Brilliant Green (229).
- 2 per cent. New Methylene Blue BB (230).
- 2 per cent. Tannin Orange R (231).
- 2 per cent. Victoria Blue B (232).
- 1 per cent. Rhodamine (233).
- 1 per cent. Brilliant Phosphine (234).
- 2 per cent. Acridine Red (235).
- $\frac{1}{2}$  per cent. Irisamine (236).

Test five of these colors for fastness to light, washing, and crocking (see Exps. 57, 58, and 66).

RECORD OF RESULTS OF TESTS.

Tests.		1	2	3	4	5
Light .....						
Washing.	Soap liquor.....					
	White wool.....					
	White cotton .....					
Crocking .....						

**Experiment 81. Principal Basic Dyes on Silk.** — Use a dye-bath containing 5 per cent. of soap and slightly acidify by the addition of sufficient acetic acid. Enter the silk at about 100° F., and gradually raise to 190° F., and dye at that temperature for one-half hour. Wash well, and brighten by passing the dyed skeins through a bath of dilute acetic acid, squeezing and drying without washing. Use the following dyestuffs:

$\frac{1}{4}$  per cent. Diamond Fuchsine (237).

2 per cent. Diamond Fuchsine (238).

3 per cent. Safranine (239).

$\frac{1}{4}$  per cent. Methylene Blue (240).

2 per cent. Methylene Blue (241).

1 per cent. Auramine (242).

2 per cent. Malachite Green (243).

2 per cent. Bismarck Brown (244).

$\frac{1}{4}$  per cent. Rhodamine (245).

2 per cent. Tannin Orange R (246).

Test five of these colors for fastness to water (see Exp. 60).

RECORD OF RESULTS OF TESTS.

Tests.		1	2	3	4	5
Test to water.	Steeping water.....	.....	.....	.....	.....	.....
	White silk.....	.....	.....	.....	.....	.....
	White cotton.....	.....	.....	.....	.....	.....

#### NOTES.

**1. The Use of Basic Colors on Cotton.** — Though the basic dyes possess a strong affinity for the animal fibres, and may be dyed on these in a neutral bath without any other addition than the dyestuff itself, cotton (and the vegetable fibres in general) possesses but a very slight attraction for this class of dyestuffs. A few of the basic dyes, such as Magenta, Chrysoidine, Bismarck Brown, and Methylene Blue, will be absorbed to a certain extent



by the cotton fibre; but most of the color may be washed out with cold water, and almost entirely removed with a warm soap solution. An exception must be mentioned of certain dyes among the class of water-soluble indulines, such as Indazine, Indamine Blue, Toluylene Blue, Nigramine, New Gray, Methylene Gray and Indoin Blue, which give dyeings of considerable fastness on cotton with no other addition to the dye-bath than sodium acetate. In order to dye cotton with the basic dyes it is customary to previously mordant the material with tannin and antimony. It may be considered that a tannate of antimony is precipitated within the fibre which exhibits a strong affinity for the basic dyes and gives with them an insoluble color-lake. The affinity between the dyestuff and the antimony tannate is usually so great that it is difficult to obtain uniform colors in the dyeing, as, for instance, with Methylene Blue, Nigramine, etc. On this account it is advised not to add all of the dyestuff to the bath at once, but to dissolve it up and add the solution in several portions. Furthermore, it is best to start the dyeing at a low temperature and not to raise the temperature too rapidly. As a rule, it will not be necessary to bring the bath to the boil, as the dyeing is usually complete at about 180° F.

In the dyeing of basic colors on cotton the tannin mordant may be applied in one of two ways: (1) steeping the cotton in the solution of tannin for a comparatively long time, then squeezing and fixing with tartar emetic or other suitable salt; (2) padding with the solution of tannin, which consists in impregnating the cotton with a strong solution for a short time, then squeezing and drying or fixing first with an antimony salt. The first method is that usually employed for yarn dyeing, while the padding method is largely used in cloth dyeing and also for the production of mordanted cloth for purposes of printing. In the steeping process it has been the custom to lay the yarn down in the tannin liquor over-night, starting at a temperature somewhat under the boiling point and allowing to cool; it is a question as to whether the cotton will absorb much more tannin in this time than in a couple of hours. From experiments which have been performed on this

point it would seem that by entering the cotton at a temperature just under the boil and allowing it to steep in the cooling liquor for about 2 hours it will absorb about as much tannin as it would if the steeping was continued for 10 to 12 hours. It is best to start the steeping at a temperature near the boiling point, chiefly for the purpose of driving out air bubbles from the fibre and causing better penetration of the tannin solution. The higher temperature does not appear to influence the actual absorption of the tannin itself by the cotton, as more is absorbed from a cooling bath than from one in which a high temperature is maintained. The strength of the tannin bath should be based on the amount of coloring-matter to be subsequently used, it being customary to take about twice as much tannin (as tannic acid and corresponding amounts of tannin extracts in accordance with the percentage of tannic acid present) as dyestuff; that is, if a color is to be obtained requiring about 2 per cent. of dyestuff, about 4 per cent. of tannin should be used for mordanting. Where light shades are being dyed it is not customary to preserve the tannin bath, but for dark heavy shades, where baths containing 4 to 10 per cent. of tannin are being used, it is best to use the baths continuously, freshening the standing bath each time with 3 to 4 per cent. of tannin. The amount of liquor used in the mordanting bath should not be more than 15 to 16 times the weight of the cotton; that is, each pound of cotton should have about 2 gallons of water for mordanting; if a greater amount of water is used, the proportion of tannin absorbed by the fibre will be lessened and a correspondingly larger amount of tannin will have to be used. The water employed in the mordanting bath should be as free from iron as possible if the tannin is to be fixed with antimony; for iron present in even a slight trace will alter the color of the dyeing considerably, especially in the case of pale shades. If the water does contain any iron, a small amount of hydrochloric acid should be added which will hold the iron tannate in solution and prevent it from contaminating the fibre. Hard water, that is, a calcareous water, unless of very considerable hardness, is not especially detrimental for use with tannin;

if there is much lime present, it may result in the precipitation of some of the mordant in the form of tannate of lime. Such water may be best corrected by the addition of sufficient acetic acid to give a slight acid reaction to the bath previous to the addition of the tannin. After the steeping in the tannin is completed, the cotton should be well squeezed or wrung out to remove the excess of liquor; in practice this may be best accomplished by hydro-extraction. It is not advisable to rinse the cotton after removal from the tannin bath, as this will only result in redissolving some of the absorbed tannin, and the residual liquor in the cotton will still be a solution of tannin; so that the rinsing does not serve the purpose of removing such residual liquor, but only results in the lessening of the mordant. There does not appear to be much difficulty attached to the uneven squeezing or wringing of the mordanted cotton leading to uneven results in the subsequent dyeing; probably if the tannin, through one cause or another, is distributed very unevenly through the cotton, there may result uneven dyeing, yet under ordinary conditions particular caution does not have to be taken in the even squeezing of the wet cotton.

After the mordanted cotton is squeezed the next operation is to pass through a fixing bath containing tartar emetic or other suitable salt of antimony. The fixing is complete in from 15 to 30 minutes and a cold bath is used; the amount of tartar emetic necessary is about one-half that of the tannin used; in other words, it is about equivalent to the amount of dyestuff to be used. For other antimony salts, corresponding amounts must be used (see previous section). The reaction in the fixing bath consists of the formation of antimony tannate in the fibre, and it should be so adjusted that all of the tannin present is thus combined; this reaction necessitates, of course, the liberation in the bath of the acid previously combined with the antimony — with tartar emetic there would be liberated tartaric acid. On this account, when using the fixing liquor as a standing bath, it will be necessary to add sufficient soda ash from time to time to neutralize the acidity, otherwise the tannate of antimony will be dissolved from the



fibre. When the fixation of the tannin mordant is completed the cotton must be thoroughly washed for the purpose of removing all uncombined tartar emetic or tannin; if excess of either of these is present in the fibre when it is passed into the dye-bath it will result in the loss of coloring-matter and probably lead to streaked and imperfect dyeing. After the mordanting and fixing operations are finished too long a time should not elapse before the dyeing, for if the mordanted cotton is exposed for any length of time to the air and light the exposed parts will turn somewhat brownish and after dyeing will appear duller. If the dyeing cannot be carried out the same day as the mordanting and fixing, the material should be covered with a moistened cloth.

In some cases in order more thoroughly to combine any excess of tartar emetic in the fibre after fixing, the cotton is passed back into the tannin bath (usually a rather weak one). This "back-tanning" may also be done after dyeing; it also appears to give colors which are faster to washing. As antimony compounds are of a poisonous nature they should be thoroughly fixed in the fibre, as otherwise blood poisoning might result from fabrics worn next the skin. In certain cases (as with Victoria Blue and Methylene Blue) in order to obtain even shades it is necessary to wash the cotton after fixing for 15 to 30 minutes in a warm soap bath (containing about 2 ounces soap to 10 gallons of water), and afterwards wash in fresh water. This treatment usually produces clearer and more even shades.

In the dyeing it is best to start the bath cold, using 25 to 30 times as much water as cotton (one pound of cotton would therefore require about 4 gallons of water), and adding first about 1 to 1½ per cent. of acetic acid; this serves the purpose of correcting any hardness in the water and thus prevents any precipitation of coloring-matter, and also makes the bath slightly acid which avoids too rapid an exhaustion of dyestuff in the dyeing. After the acid has been placed in the bath a portion of the color solution is added, and then the cotton is entered and worked for about 10 minutes; the material is then lifted, and a further portion of the dyestuff solution added, the bath being heated to about 100° F.



Finally the rest of the color is added and the bath is raised to about 140 to 160° F., and the dyeing completed. In place of acetic acid, an addition of about 3 per cent. of aluminium sulphate or 5 per cent. of alum may be made. In the case of certain basic colors the dyeing is finished by raising the temperature of the bath to the boil, as with Naphthindone.

When the tannin mordant is to be fixed with iron instead of antimony, where dark colors are to be employed, the fixing bath is made up with 3 to 5 per cent. of copperas, or consists of pyrolignite of iron at 3 to 4° Tw. The bath is employed cold, and it is well to add a small quantity of chalk (calcium carbonate) to prevent the accumulation of acid (from the acid combined with the iron salt and which will be liberated when the iron combines with the tannin to form tannate of iron); about 2 to 4 per cent. of chalk will be all that is necessary. Dyeings on an iron-tannin mordant are not so fast as those on an antimony-tannin base, so the process of fixing with iron is sometimes modified by first fixing with antimony and subsequently with iron, or even by saddening with an iron liquor after the dyeing is finished.

In some cases increased fastness to washing for basic dyeings may be obtained by giving the dyed goods a passage through the tannin bath (the old tannin liquor may be used), wringing out, and then passing through the tartar emetic bath again and washing well.

Certain of the basic colors, such as Naphthindone and Irisamine, may be dyed on cotton directly without a mordant. The dye-bath is prepared with 3 to 5 pounds of salt per 10 gallons of liquor according to the depth of shade; the dyeing is started at 110° F., and the bath is slowly brought to the boil; after dyeing the goods are simply wrung out as evenly as possible and dried. This method is not often practiced, as the colors obtained are not very fast to washing or light.

For the production of bright pinks on cotton certain basic dyes, like Irisamine, may be used on a mordant of Turkey-red oil. The yarn is mordanted in lots of 1 pound each in a liquor containing 1 part Turkey-red oil and 2 parts of water, and after each lot is

mordanted about 1 pint of such a mixture is added afresh. After mordanting, the yarn is wrung out well, straightened and dried, after which the same treatment is repeated. The dyeing is conducted in a cold concentrated bath with addition of some acetic acid, the color solution being added in several portions. In order to produce level shades it is necessary that the yarn be wrung out as evenly as possible and that the mordanting be repeated several times.

**2. List of the Principal Basic Dyes.** — The basic dyes do not include such a large list as the acid dyes, though the apparent number is considerably increased by the fact that the same dye-stuff is frequently given a variety of different names, and furthermore, a number of mixtures are marketed under specific names. Some of these dyes are more adapted to the dyeing of silk than of cotton, and vice versa; this can only be determined by reference to the specific properties of the individual dyestuffs.

(a) RED:

Acridine Red.	Magenta Scarlet.
Acridine Scarlet.	Maroon.
Brilliant Rhoduline Red.	Neutral Red.
Cardinal.	Neutral Scarlet.
Cerise.	New Magenta.
Diamond Fuchsine.	Parafuchsine.
Diamond Magenta.	Patent Rhodamine.
Fuchsine.	Pyronine.
Grenadine.	Rhodamine.
Induline Scarlet.	Rhodine.
Irisamine.	Rhoduline Red.
Isorubine.	Rosazeine.
Janus Bordeaux.	Rubine.
Janus Red.	Safranine.
Magenta.	

(b) ORANGE.

Acridine Orange.	Homophosphine.
Azo Phosphine.	New Acridine Orange.
Brilliant Phosphine.	New Phosphine.
Canelle.	Paraphosphine.
Chrysoidine.	Patent Phosphine.
Coriphosphine.	Phosphine.
Flavinduline.	Tannin Orange.

(c) YELLOW.

Acridine Yellow.  
Auracine.  
Auramine.  
Benzoflavine.  
Janus Yellow.

Leather Yellow.  
Rheonine.  
Thioflavine T.  
Xanthine.

(d) GREEN.

Azine Green.  
Bengal Green.  
Brilliant Green.  
Capri Green.  
China Green.  
Diamond Green.  
Diazine Green.  
Ethyl Green.  
Janus Green.

Malachite Green.  
Methyl Green.  
Methylene Green.  
New Fast Green.  
New Green.  
New Solid Green.  
New Victoria Green.  
Victoria Green.

(e) BLUE.

Azindone Blue.  
Basle Blue.  
Bavarian Blue.  
Bengal Blue.  
Bleu de Lyon.  
Bombay Blue.  
Brilliant Blue.  
Brilliant Cresyl Blue.  
Brilliant Diazine Blue.  
Brilliant Metamine Blue.  
Brilliant Victoria Blue.  
Capri Blue.  
China Blue.  
Cotton Blue.  
Cotton Light Blue.  
Cresyl Blue.  
Crystal Fast Blue.  
Dark Blue.  
Diazine Blue.  
Diphen Blue.  
Diphenylamine Blue.  
Ethyl Blue.  
Ethylene Blue.  
Excelsior Cotton Blue.

Fast Blue.  
Fast Blue for cotton.  
Fast Cotton Blue.  
Fast Marine Blue.  
Fast New Blue.  
Gentianine.  
Helvetia Blue.  
Indamine Blue.  
Indanil Blue.  
Indazine.  
Indigen.  
Indigo Blue.  
Indoine.  
Indoine Blue.  
Indol Blue.  
Indone Blue.  
Indophenine Blue.  
Janus Blue.  
Janus Dark Blue.  
Light Blue.  
Marine Blue.  
Metaphenylene Blue.  
Methyl Blue.  
Methyl Cotton Blue.

## (e) BLUE. — Continued.

Methyl Indone.	Opal Cotton Blue.
Methyl Light Blue.	Paper Blue.
Methyl Water Blue.	Paraphenylene Blue.
Methylene Blue.	Peacock Blue.
Methylene Dark Blue.	Phenine Blue.
Methylene Indigo.	Phenylene Blue.
Muscarine.	Pure Blue.
Naphthindone.	Rhoduline Blue.
Naphthol Blue.	Rhoduline Sky Blue.
Neutral Blue.	Setocyanine.
Neutral Peacock Blue.	Setoglaucine.
New Blue.	Setopaline.
New Diamond Indigo Blue.	Solid Blue.
New Fast Blue.	Thionine Blue.
New Indigo Blue.	Toluidine Blue.
New Metamine Blue.	Toluylene Blue.
New Methylene Blue.	Turkey Blue.
New Solid Blue.	Turquoise Blue.
New Victoria Blue.	Victoria Blue.
Night Blue.	Victoria Night Blue.
Nile Blue.	Water Blue.

## (f) VIOLET.

Brilliant Rhoduline Purple.	Methylene Violet.
Brilliant Violet.	Neutral Violet.
Cresyl Fast Violet	Paraphenylene Violet.
Crystal Violet.	Primula.
Dahlia.	Red Violet.
Ethyl Purple.	Regina Violet.
Ethyl Violet.	Rhoduline Heliotrope.
Fast Neutral Violet.	Rhoduline Violet.
Giroflé.	Rosolane.
Heliotrope.	Rubine Violet.
Irisamine.	Soda Violet.
Iris Violet.	Tannin Heliotrope.
Methyl Violet.	Violet.
Methylene Heliotrope.	

## (g) BROWN.

Bismarck Brown.	Manchester Brown.
Brown extra soluble.	Tannin Brown.
Cutch Brown.	Vesuvine.
Janus Brown.	



(h) BLACK.

Coal Black.	Methylene Gray.
Diazine Black.	New Fast Gray.
Fast Black.	New Gray.
Fast Gray.	New Methylene Gray.
Janus Black.	Nigrisine.
Janus Gray.	Paper Black.
Jute Black.	Silk Gray.
Jute Coal Black.	Straw Black.
Leather Black.	

SAMPLES.

227. Bismarck Brown on tannin-antimony mordant on cotton.
228. Safranine on same.
229. Brilliant Green on same.
230. New Methylene Blue BB on same.
231. Tannin Orange R on same.
232. Victoria Blue B on same.
233. Rhodamine on same.
234. Brilliant Phosphine on same.
235. Acridine Red on same.
236. Irisamine on same.
237. Diamond Fuchsine ( $\frac{1}{4}$  per cent.) on silk.
238. Diamond Fuchsine (2 per cent.) on same.
239. Safranine on same.
240. Methylene Blue ( $\frac{1}{4}$  per cent.) on same.
241. Methylene Blue (2 per cent.) on same.
242. Auramine on same.
243. Malachite Green on same.
244. Bismarck Brown on same.
245. Rhodamine on same.
246. Tannin Orange R on same.

QUIZ 12.

327. Name five of the principal basic dyes applied to cotton. What colors do they give?
328. What degree of fastness did you find for the colors selected when tested to light, washing, and crocking?
329. Name five of the principal basic dyes employed on silk, with the character of the colors they give on this fibre. How do these compare in brightness with corresponding acid dyes?

330. What was the result of your tests for fastness to water of the basic dyes on silk? How do they compare in this respect to the corresponding acid dyes which were tested?

331. Does cotton possess any direct attraction for the general class of basic dyes? Are there any exceptions to this general rule?

332. In order to dye cotton with the basic dyes what previous treatment is necessary? Of what does the mordant consist? How does this mordant react with basic dyes?

333. Is the attraction of the basic dyes for antimony tannate strong or weak, and how is this shown in the dyeing?

334. What precautions are recommended in the dyeing of basic colors on cotton in order to obtain level shades?

335. In what two ways may the tannin mordant be applied to cotton, and for what classes of material is each most adaptable?

336. What is meant by "padding" and how is this done? What kind of a solution is used for padding?

337. Give the process of mordanting cotton with tannin by the steeping process. What length of time is required for this process?

338. Why should the mordanting bath be started near the boiling point? Under what conditions is the greatest amount of tannin taken up by the cotton?

339. How should the strength of the tannin bath be regulated? How much tannin should you use for the dyeing of 50 pounds of cotton where the color calls for  $1\frac{1}{2}$  per cent. of dyestuff?

340. Is it possible to employ the tannin solution as a standing bath? How much tannin should be used for freshening?

341. How many pounds of sumac extract containing 20 per cent. of tannin should be used for mordanting 75 pounds of cotton yarn which are to be dyed with 3 per cent. of coloring-matter?

342. How much water should be used in the mordanting bath in proportion to the weight of the cotton to be mordanted?

343. How many gallons of tannin liquor would be required for the mordanting of 75 pounds of cotton yarn? If gambier containing 35 per cent. of tannin is to be used for mordanting, how much should be taken if the cotton is to be dyed with 2 per cent. of dyestuff?

344. What character of water should be used in the tannin bath? If the tannin is to be fixed with antimony why should the water be free from iron?

345. If water containing iron has to be used for mordanting with tannin, how may it be corrected? Explain the action of the substance used for correcting.

346. Can calcareous water be used in the tannin bath? What action takes place between the lime and the tannin? If the water is highly calcareous how may it be corrected for use in the tannin bath?

347. After the cotton has been steeped in the tannin solution should it be rinsed? Why?

348. In the wringing out of the cotton after mordanting with tannin do particular pains have to be taken in order to squeeze out the liquor evenly?

349. How is the tannin fixed on the cotton after mordanting? Why is this necessary?

350. How is the fixing bath of tartar emetic prepared? How long does the fixing require and what temperature? How much tartar emetic should be used?

351. If 75 pounds of cotton are to be dyed with  $1\frac{1}{2}$  per cent. of a basic dyestuff, how many pounds of tartar emetic should be used for fixing the tannin?

352. How much antimony salt would be required in the above example? How much antimony oxalate and how much antimonine?

353. What reaction takes place in the fixing bath of tartar emetic? With antimony fluoride? With antimony oxalate? With antimonine?

354. If the antimony fixing liquor is used as a standing bath what addition is necessary and why?

355. After fixing the tannin with antimony why is it necessary to thoroughly wash the cotton? What defects would be liable to occur if this were not done?

356. Should the dyeing of basic colors on cotton take place immediately after the mordanting and fixing, and why?

357. If the dyeing on the mordanted cotton cannot be done on the same day what should be done with the material?

358. What is meant by "back-tanning"? How is this done and what is its purpose?

359. If any free antimony fixing compound is left in the goods after dyeing material used for clothing, what defect is liable to arise?

360. In order to insure clear even shades with Victoria Blue how should the cotton be treated after fixing?

361. In dyeing cotton with basic colors at what temperature should the bath be started, how much water should be used, and what addition should be made before the color is added? What is the purpose of this addition?

362. In what manner should the basic dyestuff be added to the dye-bath, and how should the temperature of the bath be regulated?

363. Instead of adding acetic acid to the dye-bath for basic colors, what other additions may be made? Are basic colors ever dyed at the boil?

364. When may the tannin mordant on cotton be fixed with iron in place of antimony? What salts of iron may be used? What is the color of iron tannate?

365. In fixing tannin with an iron salt how may acidity in the fixing bath be neutralized? From what does this acidity arise?

366. Are dyeings on an iron-tannin mordant as fast as if antimony were used for fixing? How may the process of fixing tannin with iron be modified in order to obtain faster dyeings?

367. What basic colors may be applied to cotton without a mordant? How is the dye-bath prepared in such a case? Are the colors fast?

368. How may bright pinks be obtained on cotton with an oil mordant? What oil is used? How is the mordanting conducted? How is the dyeing carried out and in what kind of a bath? What precautions are necessary in order to obtain level colors?



## SECTION XIII.

### APPLICATION OF SUBSTANTIVE DYES TO COTTON.

**Experiment 82. General Method of Dyeing Cotton.** — These dyes are usually applied to cotton in a neutral bath containing either common-salt or glaubersalt; hence, the name of “salt” or “direct cotton” colors for this class of dyes. Dye a skein of cotton yarn in a bath containing 300 cc. of water, 20 per cent. of common-salt, and 1 per cent. of Congo Red; enter at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour; then wash well and dry (247). The bath does not exhaust very well, but by adding more salt towards the end of the dyeing a better degree of exhaustion may be obtained, although the colors are not apt to be so fast to washing. The use of the common-salt (or of glaubersalt) in the bath is to increase the exhaustion and give better penetration of the color through the fibre. The substantive dyes give good colors on cotton, many of them being fast to light, acids, and alkalies, though some of them are changed by treatment with acids, as is the case with Congo Red. To show this action, dip a few strands of the dyed yarn plaited together into a dilute solution of sulphuric acid; it will be found that the red color is changed to a bluish black. The red color may be brought back by treatment with alkalies, which may be shown by dipping a portion of the above sample in a dilute solution of soda ash. Do this carefully and then wash well so that the sample (248) will show one-half discolored and the other half red. The chief defect of the substantive dyes however, on cotton, is their liability to bleed on washing in hot water or soap solutions. To show this action, make up two plaited samples from the dyed skein together with white cotton yarn; boil one of these in plain water for 15 minutes, then squeeze and dry (249), when it will be found that the color has bled into the white



yarn. Scour the other sample in a warm dilute soap bath, then wash in fresh water and dry (250), and note if the color has bled or not into the white.

**Experiment 83. Influence of the Amount of Salt in the Dye-bath.** — Dye skeins of cotton yarn in baths containing 3 per cent. of Chicago Blue and the respective amounts of common-salt as given below; enter at 160° F., bring to the boil, and dye at that temperature for one-half hour, then wash well and dry (251, 252, 253, 254).

- |                              |                                |
|------------------------------|--------------------------------|
| (1) Use no salt.             | (3) Use 20 per cent. of salt.  |
| (2) Use 5 per cent. of salt. | (4) Use 100 per cent. of salt. |

Compare the depth of color obtained on the several skeins and determine what influence, if any, the amount of salt has on the color taken up by the fibre.

**Experiment 84. Use of Soda Ash.** — Very often a better degree of exhaustion of the dye-bath and a greater fastness of the color to washing may be obtained by dyeing substantive colors in a bath made slightly alkaline with soda ash. Dye a skein of cotton yarn in a bath containing 20 per cent. of salt, 1 per cent. of soda ash, and 2 per cent. of Columbia Green; enter at 160° F., gradually bring to the boil, and dye at that temperature for one-half hour. Wash well and dry (255). In place of using soda ash, which is a strong alkali, milder alkaline salts such as sodium phosphate or sodium silicate may be used.

**Experiment 85. Use of Soap.** — This method is somewhat similar to the preceding, except that soap is used for making the bath alkaline; it is also supposed that this makes the color somewhat faster to washing with soap. Dye a skein of cotton yarn in a bath containing 5 per cent. of soap and 2 per cent. of Columbia Green; enter at 160° F., gradually bring to the boil, and dye at that temperature for one-half hour; wash well and dry (256). Salt cannot be used in the bath, as it precipitates the soap. For comparison dye another skein of cotton yarn in a bath with 20 per cent. of salt and 2 per cent. of Columbia Green in the usual

manner; wash well and dry (257). Compare the colors obtained on these skeins with the one in the previous experiment; also test them for fastness to washing and note if the method of dyeing in the alkaline or soap baths has increased the fastness of the color to any extent.

**Experiment 86. After-treatment with Chrome.** — This treatment is for the purpose of increasing the fastness of certain substantive dyes to washing and acids; it also deepens the color, as a rule, to quite a degree, and in some cases causes a considerable change in the tone of the color. Dye two skeins of cotton yarn together in a bath containing 20 per cent. of common-salt and 2 per cent. of Chromanil Brown 2G; enter at 160° F., gradually raise to the boil, and dye at that temperature for one-half hour; wash well, and set one of the skeins aside for comparison (258). Take the second skein and pass into a bath containing 2 per cent. of chrome; boil for 15 minutes, then wash well and dry (259). Compare the colors obtained on each of the skeins and thus note the effect of the after-chroming on the color. Make tests on both skeins for fastness to washing and perspiration; also test the colors for their fastness to light.

**Experiment 87. After-treatment with Bluestone.** — This treatment is usually for the purpose of giving an increased fastness to light; the color is also generally considerably altered in tone by the treatment. Dye two skeins of cotton yarn together in a bath containing 20 per cent. of common-salt and 2 per cent. of Diamine Blue RW; enter at 160° F., gradually raise to the boil, and dye at that temperature for one-half hour; wash well, and set one of the skeins aside for comparison (260). Pass the second skein into a fresh bath containing 3 per cent. of bluestone and 3 per cent. of acetic acid; work for 15 minutes at a temperature of 180° F., then wash well and dry (261). Compare the color on the two skeins, and make a test on each for its fastness to light (262, 263).

**Experiment 88. Dyeing in a Cold Bath.** — Many of the substantive dyes are taken up by cotton from a cold bath almost as well as from a hot bath, and these become very useful in



cases where it is not desirable to employ a very hot liquor in the dye-bath. Dye five test-skeins of cotton yarn with the following dyestuffs respectively in baths containing 20 per cent. of common-salt and 2 per cent. of the dyestuff, enter cold and dye (without heating) for three-quarters hour; then wash and dry (264, 265, 266, 267, 268). It is necessary to have the yarn very well boiled-out for this method of dyeing, as otherwise it will be difficult to obtain good penetration of the coloring-matter into the fibre. It is also well to add to the dye-bath a small amount of Turkey-red oil in order to increase the penetration. Use the following dyestuffs:

Erika BN.	Chrysophenine.	} (Ber.)
Brilliant Orange G.	Chicago Blue 6B.	
	Heliotrope 2B.	

For the dyeing of light shades soap is often added to the bath, as this helps the wetting-out of the cotton; for heavy shades, besides Turkey-red oil, there may also be added a small amount of soda ash to give a better exhaustion of the bath.

**Experiment 89. Shading Substantive Dyes with Basic Dyes.** — Substantive dyes act as mordants toward basic dyes, on which account the latter may be employed for purposes of topping or shading the former. According to the depth of the substantive dyeing, from  $\frac{1}{4}$  to  $\frac{1}{2}$  per cent. of basic dye may be fixed with considerable fastness to washing. Almost any substantive dye may be used as the bottom color, and almost any basic dye may be employed for topping. The dyeing with the substantive color is carried out in the usual manner, while the topping with the basic color is done in a fresh cold bath, either with or without the addition of a small amount of acetic acid. The method is used for the purpose of giving increased depth of color as well as increased brightness; for the substantive colors, as a rule, are neither very intense nor very bright. In some cases the fastness of the color to washing and light is also increased. Dye five test-skeins of cotton yarn in the usual manner with 2 per cent. of Chrysophenine (269); wash well and top the five skeins as



follows in cold baths containing  $\frac{1}{4}$  per cent. of the respective dyestuffs:

- |                      |                 |
|----------------------|-----------------|
| (1) Methylene Blue.  | (3) Rhodamine.  |
| (2) Methyl Violet.   | (4) Auramine O. |
| (5) Malachite Green. |                 |

Enter cold and dye at that temperature for one-half hour; wash well and dry (270, 271, 272, 273, 274).

Dye a skein of cotton yarn in the usual manner with 2 per cent. of Chicago Blue 6B; wash well (275); and top as in the foregoing test with  $\frac{1}{4}$  per cent. of Methylene Blue; wash well and dry (276).

Dye a skein of cotton yarn in the usual manner with 3 per cent. of Diamine Scarlet 3B; wash well (277), and top as before with  $\frac{1}{2}$  per cent. of Rhodamine (278).

In each case preserve a sample of the skein dyed with the substantive color alone and compare it with the topped sample.

#### NOTES.

1. **The Substantive Dyestuffs.** — These coloring-matters were first discovered in 1884 in the dyestuff known as Congo Red by Boettiger. They are distinguished by the common property of dyeing the vegetable fibres in full and comparatively fast shades without the intervention of mordants; they also dye the animal fibres, wool and silk. Their chief application, however, is to cotton. At present there are several distinct classes of substantive dyes as far as their chemical constitution is concerned, but for the most part they are derived more or less directly from the parent substance benzidine, and are characterized by being "tetrazo" compounds; that is to say, their molecule contains the azo group  $N = N$  twice. As benzidine is a diamine compound (that is, contains the diamine group,  $NH_2$ , twice), these colors are also known as the "diamine" colors. The nature of the dyeing process with regard to the substantive colors on cotton is not as yet thoroughly understood; unlike the dyeing of the acid and basic colors, there appears to be no reason for assuming that a chemical reaction occurs between the fibre and the dyestuff. The substantive dyes as a rule are very soluble in water, and conse-

quently the dye-baths are seldom completely exhausted even when relatively small amounts of the coloring-matter are used. Cotton which has been dyed with a substantive color will also usually bleed, or have some of its color extracted again when boiled in fresh water; and this extraction of color may be successively repeated until a large part of the dyestuff has been removed from the fibre. Again, the amount of coloring-matter which can be taken up by the cotton fibre appears to be rather limited, on which account very heavy, dense shades, as a rule, cannot be obtained with the substantive colors on cotton. By the addition of salt to the dye-bath the solubility of the coloring-matter in the water is lessened, and consequently more of the color is forced on the cotton; this condition is also favored by employing as "short" a bath as possible, that is, one containing a minimum amount of dye liquor. Either common-salt (sodium chloride) or glauber-salt (sodium sulphate) may be used in the dye-bath, though the former is mostly used, as it is anhydrous and does not require such a large amount to be added. In common practice about 20 per cent. of salt is used in the bath, though when it is desired to obtain heavy shades or to get a better degree of exhaustion larger amounts of salt may be used, even to as high as 100 per cent. on the weight of the cotton being dyed. If too great an amount of salt is added there will be danger of some of the dyestuff being precipitated or "salted out"; this, however, as a rule will not occur until about 1 pound of salt per gallon of solution has been added, an amount which will hardly ever be used in practice. When the dye liquor, however, is employed as a standing bath, care must be had that in the successive additions of dyestuffs and salt the accumulation of the latter in the bath does not become too great. In order to control this amount the density of the liquor should be determined with a hydrometer. For light shades, as a rule, but little salt is used, and as only a slight proportion of color remains in the bath, the liquors are seldom kept for further use. For medium shades the best density of the dye liquor is about 2° Tw., and for dark shades from 4 to 6° Tw. In determining the density of the liquor with the hydrom-

eter, a small portion should be taken from the vat and allowed to cool before being tested. When dyeing in baths containing a large amount of salt, it is best not to add the salt until towards the end of the operation, and the goods after coming from the dye-bath should be well rinsed in fresh water, otherwise the salt may crystallize in the material and afterwards be more difficult to remove. Increased exhaustion of the dye-bath may also be obtained by using vats heated with closed steam coils, as the introduction of live steam into the bath considerably dilutes it. Increased exhaustion is also obtained by allowing the color to feed on the cotton from a cooling bath; that is to say, the cotton should not be taken from the bath at a boil, but the steam should be turned off and the bath allowed to cool down with the cotton in it. As the substantive colors are so soluble in water and exhaust so poorly, it will seldom occur that they will dye unevenly; if such, however, does happen, they may be easily levelled by continued working in a boiling bath.

Many of the substantive dyes appear to work somewhat better when the bath is made slightly alkaline by the addition of soda ash, sodium phosphate, sodium silicate, borax, soap, etc. Just what is the action of the alkali in this case is uncertain; it probably aids in the penetration of the coloring-matter into the fibre. For light shades it is sometimes beneficial to add Turkey-red oil to the bath. In preparing the dye-bath in practice, it is best to first add the alkali (if such is used), then the color solution, and finally the salt.

The substantive colors should be dissolved in boiling water, and if possible, water from condensed steam should be used. If the water to be used for dissolving the dyestuff is calcareous, it is best to first boil the water up with an amount of soda ash equivalent to the weight of the dyestuff to be dissolved. After dissolving the color, the solution should be strained through a piece of cotton cloth or fine sieve. When the dyestuff is added in an undissolved condition directly to the dye-bath, some soda ash should first be added, the bath boiled up, and then the dyestuff added, after which the salt is added.

Where hard water must be used in dyeing substantive colors



it should always first be corrected by treatment with a suitable amount of soda ash. With certain colors which are especially sensitive to hard water, it is recommended to dye with addition of 2 to 4 per cent. of acid potassium oxalate, the amount depending on the hardness of the water and the quantity of dyestuff to be used; it should be noted that an excess of the oxalate is injurious, causing the color to exhaust badly and giving dull shades.

It is a mistake to suppose that the substantive dyes require a vigorously boiling bath for dyeing; while it is true that a boiling bath will give a better penetration of color, it is also a fact that the amount of color absorbed by the cotton is greater when the temperature of the bath is under the boil, and it has already been pointed out that a better degree of exhaustion is obtained by allowing the goods to remain for some time in the cooling bath. When desirable, most of the substantive colors may be dyed at moderate temperatures, and even cold. In such cases it is best to add to the bath some soap or Turkey-red oil in order to obtain better penetration of the coloring-matter. When dyeing in a cold bath it is sometimes recommended to mix the dyestuff first with its own weight of caustic soda solution (78° Tw.), then dissolve in a sufficient quantity of hot water, and add this solution to the dye-bath along with a little soap.

For dyeing light shades for each 100 pounds of cotton yarn about 200 gallons of water should be used, while for dark shades the amount of water should be limited to about 130 gallons. For dark shades, especially where only one dyestuff is used in the color, the yarn can usually be entered at the boil; for lighter shades, and where several dyes may be used in combination, it is best to enter the cotton at 140 to 160° F., and gradually raise to the boil. If any tendency towards unevenness is observed, it is best to add only a part of the salt to the bath at first and reserve the rest to be added near the end of the dyeing. Yarn which has been dyed in light shades is not generally rinsed after dyeing unless alkali has been used in the bath; but where heavy shades are obtained the yarn should always be well rinsed in order to remove all excess of residual dye liquor and salt solution.



**SAMPLES.**

- 247. Congo Red on cotton, showing method of dyeing.
- 248. Congo Red tested with acid and alkali.
- 249. Congo Red tested for bleeding.
- 250. Congo Red tested for fastness to washing.
- 251. Chicago Blue dyed with no salt.
- 252. Chicago Blue dyed with 5 per cent. salt.
- 253. Chicago Blue dyed with 20 per cent. salt.
- 254. Chicago Blue dyed with 100 per cent. salt.
- 255. Columbia Green dyed with soda ash.
- 256. Columbia Green dyed with soap.
- 257. Columbia Green dyed with salt alone.
- 258. Chromanil Brown before treatment.
- 259. Chromanil Brown treated with chrome.
- 260. Diamine Blue RW before treatment.
- 261. Diamine Blue RW treated with bluestone.
- 262. Untreated sample exposed to light.
- 263. Treated sample exposed to light.
- 264. Erika BN dyed in a cold bath.
- 265. Brilliant Orange G dyed in a cold bath.
- 266. Chrysophenine dyed in a cold bath.
- 267. Chicago Blue 6B dyed in a cold bath.
- 268. Heliotrope 2B dyed in a cold bath.
- 269. Chrysophenine (2 per cent.) as a bottom color.
- 270. Topped with Methylene Blue.
- 271. Topped with Methyl Violet.
- 272. Topped with Rhodamine.
- 273. Topped with Auramine.
- 274. Topped with Malachite Green.
- 275. Chicago Blue 6B as a bottom color.
- 276. Topped with Methylene Blue.
- 277. Diamine Scarlet 3B as a bottom color.
- 278. Topped with Rhodamine.

**QUIZ 13.**

369. In what character of bath are the substantive colors usually applied to cotton? Why are these dyes sometimes called "salt" or "direct cotton" colors?

370. Does the dye-bath with Congo Red exhaust well? How may the degree of exhaustion be influenced? In the latter case are the colors obtained as fast?

371. What is the function of the common-salt in the dye-bath when using substantive colors? What other neutral salt may be used?

372. What is the general fastness of the substantive dyes on cotton? What is their chief defect?

373. What takes place when the color obtained with Congo Red is treated with acid? And subsequently treated with soda ash?

374. Was the dyeing with Congo Red fast to bleeding and to the washing test? How were these tests made?

375. What do you consider the influence of varying amounts of salt in the bath in dyeing substantive colors on cotton?

376. Why is soda ash used at times in dyeing substantive colors? What other alkalies in place of soda ash may be used?

377. Why is it recommended at times to add soap to the dye-bath with substantive colors? How much soap is used? Can salt be used in such a bath, and why?

378. What is the purpose of after-treating certain substantive colors with chrome? What effect has the process on the tone of the color?

379. How is the after-treatment with chrome carried out? What difference did you find in the fastness to washing, perspiration, and light between the treated and the untreated dyeings? How did you conduct these tests?

380. What is the purpose of after-treating some substantive colors with bluestone? How is the process conducted? What effect has the treatment on the tone of the color? Did you find any marked difference in the fastness to light of the two samples tested?

381. Can substantive dyes be applied to cotton in a cold bath? How is this done? Why is Turkey-red oil added and why should the yarn be well boiled out?

382. For dyeing light shades in a cold bath what addition may be made to the bath? For heavy shades what additions are made?

383. What color does the dyestuff Erica give? What color does Chrysophenine give?

384. How do substantive dyes act towards basic dyes? What is meant by "topping" a color?

385. How much basic color may be fixed on a substantive dyeing? How is the topping with the basic color carried out?

386. What is the purpose of topping a substantive dyeing with a basic color? Describe the results you obtained on topping Chrysophenine with various basic colors.

387. What is the effect of topping Chicago Blue with Methylene Blue? Of topping Diamine Scarlet with Rhodamine?

388. When were the substantive dyes first introduced and by whom? What was the first dye discovered?

389. What is the distinguishing characteristic of the substantive dyes as a class? To what fibre are they chiefly applied?

390. From what parent substance are most of the substantive dyes derived? By what chemical group are they characterized?

391. What is meant by a "tetrazo" compound? By a "diamine"?

392. Is the nature of the dyeing process with substantive colors well understood? How does it differ from that of the acid and basic dyes?

393. What may be said as to the solubility of the substantive dyes? How does this affect their exhaustion from the dye-bath? Why do their colors usually bleed from cotton when steeped or boiled in water?

394. Can very heavy shades be obtained on cotton with the substantive dyes? Why?

395. What conditions favor a better degree of exhaustion of the substantive dye-bath?

396. What is the danger of adding too great an amount of salt to the bath with substantive colors? What is the maximum amount of salt which should ever be added?

397. In using standing baths with substantive dyes how is the concentration of the liquor controlled? What should be the density for light shades; for heavy shades?

398. When dyeing in baths containing a large amount of salt why should the goods be well washed immediately after coming from the bath?

399. In what manner should dye-vats be heated if it is desired to keep the liquor concentrated?

400. What is the effect of allowing the cotton to remain in the cooling bath when dyeing with the substantive colors?

401. Are the substantive colors liable to dye unevenly? Why? If uneven, how may the color be levelled?

402. What is the object of dyeing some substantive colors in an alkaline bath? What alkalies may be employed? What is the probable action of the alkali?

403. When dyeing substantive colors with an alkaline bath in what order should the various additions to the bath be made?

404. In dyeing light shades with the substantive colors what addition to the bath is beneficial?

405. In what manner should the substantive colors be dissolved? Should hard water be employed for this purpose?

406. What is meant by "calcareous" water? How may this water be corrected for use with the substantive dyes?

407. When substantive colors are added directly to the dye-bath how should the preparation of the bath be effected?

408. Some substantive dyes are very sensitive to hard water; what addition should be made to the dye-bath in such a case? Explain how this will neutralize the hardness of the water. Why is an excess of the chemical detrimental?

409. Do substantive colors require a vigorous boiling in dyeing? What purpose does the boiling effect? Under what conditions is the greatest amount of color absorbed?

410. May substantive colors be dyed in a cold bath? What additions to the bath should be made and for what purpose?

411. What method is recommended for dissolving the dyestuff when using substantive colors in a cold bath?

412. In dyeing light shades with substantive colors how many gallons of water should be used for 100 pounds of cotton? How many gallons when dyeing dark shades?

413. When dyeing heavy shades with a single substantive dye on cotton at what temperature may the goods be entered in the bath? What temperature is best when light shades or compound colors are being dyed?

414. If in dyeing substantive colors the shades come up uneven what is the best procedure to adopt to obtain level colors?

415. After cotton is dyed with substantive colors should it be washed? Under what conditions may the washing be dispensed with?



## SECTION XIV.

### SUBSTANTIVE DYES ON WOOL AND SILK.

**Experiment 90. General Method of Dyeing Wool.** — These colors are usually dyed on wool in a neutral bath containing either Glaubersalt or common-salt. Although the substantive colors are primarily dyes for use on cotton, nevertheless, they are being employed to a considerable extent on wool, as many of them give colors of eminent fastness. Dye a skein of woollen yarn in a bath containing 20 per cent. of Glaubersalt and 3 per cent. of Diamine Scarlet 3B. Enter at 140° F., gradually bring to the boil, and dye at that temperature for one-half hour, then wash well and dry (279). Common-salt may be used in the bath in place of Glaubersalt and has the same effect. The purpose of the addition of these neutral salts is to cause a better penetration and distribution of the coloring-matter, and also to cause a better exhaustion of the dye-bath. The substantive dyes, as a rule, are very soluble in water, and show no particular tendency to go on the fibre unevenly, hence the material may be entered at comparatively high temperatures without the danger of unevenness. Also due to its good solubility in water, the dyestuff does not give complete exhaustion in the bath. The substantive dyes do not produce as bright or as full shades on wool as the basic and acid dyes. Some of the substantive dyes, especially the reds, are very sensitive to the action of acids, their color being changed, as has been shown in the previous section, with Congo Red. The dyestuff used above, however, is fast to acid, as may be shown by moistening a small sample of the dyed yarn with a dilute solution of sulphuric acid, washing and drying (280). Also test this color for its fastness to fulling (281).

**Experiment 91. Use of Ammonium Acetate in the Dye-bath.** — The use of this salt in the dye-bath appears to give better exhaus-

tion and also to make the color faster to fulling. Dye a skein of woollen yarn in a bath containing 5 per cent. of ammonium acetate and 3 per cent. of Diamine Green G; enter at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour. Wash and dry (282). The ammonium acetate, on boiling, decomposes into ammonia (which is volatilized) and free acetic acid, and this no doubt helps in the dyeing of the wool. Ammonium acetate may be readily prepared by mixing ammonia water and acetic acid in the following proportions: 32 parts of strong ammonia water and 50 parts of acetic acid (1.031 sp. gr.) which will give a solution containing 25 parts of ammonium acetate. Diamine Green gives a color on wool which has good fastness to washing.

**Experiment 92. Dyeing in a Slightly Acid Bath.** — Some of the substantive dyes may be applied to wool in slightly acid baths in much the same manner as the acid dyes. This method is especially useful for the production of two-color effects on mixtures containing wool and cotton. Dye a skein of woollen yarn in a bath containing 20 per cent. of Glaubersalt, 4 per cent. of acetic acid, and 2 per cent. of Chrysophenine; enter at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour; wash and dry (283). Acetic acid is mostly used in this connection, as sulphuric acid is too strong and is liable to injure the color, and where cotton is present there is danger of the latter fibre being tendered by the incomplete removal of the acid, whereas acetic acid, being volatile, is easily removed. Chrysophenine gives a good yellow color on wool which is exceedingly fast to light; to show this expose a sample to light for 30 days (284).

**Experiment 93. Showing the Application of Substantive Dyes on Union Material.** — Dye a skein of union yarn (containing wool and cotton threads twisted together) in a bath containing 20 per cent. of Glaubersalt, 4 per cent. of acetic acid, and 2 per cent. of Chrysophenine; enter at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour; wash well and dry (285). It will be found that both fibres will be dyed about a

uniform color. Dye a second skein of union yarn in a bath containing 20 per cent. of Glaubersalt, 4 per cent. of acetic acid, 2 per cent. of Chrysophenine and 1 per cent. of Acid Violet. Dye in the usual manner, wash well, and dry (286). It will be found in this case that the wool has been dyed with both the yellow and violet colors, giving a resultant olive green, whereas the cotton has been dyed with the yellow and has only been slightly tinted with the violet, so that a two-color effect has been obtained.

**Experiment 94. After-treatment with Chrome.** — This treatment is for the purpose of increasing the fastness of the color to washing and light. At the same time it also causes an increase in the depth of the color. Dye two test-skeins of woolen yarn in a bath containing 20 per cent. of Glaubersalt, 10 per cent. of ammonium acetate, and 3 per cent. of Diamine Fast Red F; enter at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour. Remove one of the skeins, wash, and dry (287). Add to the dye-bath 2 per cent. of chrome, reënter the second skein and continue boiling for 20 minutes; then wash and dry (288). Compare the colors obtained on these two samples, and test each skein for its fastness to washing (289, 290).

**Experiment 95. After-treatment with Chromium Fluoride.** — This salt is sometimes substituted for chrome. It acts in much the same manner by increasing the fastness of certain colors to light and washing, and also deepening the shade. As it is not a strong oxidizing agent like chrome, it may be used at times where the latter cannot. Dye two test-skeins of woolen yarn in a bath containing 20 per cent. of Glaubersalt and 3 per cent. of Columbia Green; enter at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour. Remove one of the skeins, and wash and dry (291). Add to the dye-bath 3 per cent. of chromium fluoride, reënter the second skein and boil for 20 minutes longer; then wash and dry (292). Compare the two skeins with respect to their color and make tests on each to determine the fastness to washing (293, 294).

**Experiment 96. Example of a Substantive Dye not Coloring Wool.** — There are a few of the substantive dyes which are not



taken up by the wool fibre, especially if the bath is at a comparatively low temperature and is slightly alkaline. Dye a test-skein of woolen yarn in a bath containing 20 per cent. of Glaubersalt, 1 per cent. of soda ash, and 2 per cent. of Mikado Yellow; enter at 100° F., gradually raise to 120° F., and dye at that temperature for one-half hour; then wash well and dry (295). It will be found that the wool is hardly tinted by this color. On this account, such dyes are very useful for the dyeing of union materials where it is desirable to leave the wool undyed.

**Experiment 97. General Method of Applying Substantive Dyes to Silk.** — This fibre, like wool, will dye very readily with many of the substantive colors, yielding shades which are fast to washing and water and in many cases fast to light. Dye a skein of silk in a bath containing 10 per cent. of soap (or a boiled-off liquor bath may be employed where this is available) and 3 per cent. of Benzo Fast Scarlet; enter at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour. Wash well and dry (296). Silk may also be dyed in a slightly acid bath, as with wool. Dye a skein of silk in a bath containing 3 per cent. of Chrysophenine, 10 per cent. of Glaubersalt, and 4 per cent. of acetic acid; enter at 140° F., bring to 180° F., and dye at that temperature for one-half hour. Wash well and dry (297). As with wool, silk dyed with the substantive colors may be after-treated with chrome, etc., in order to obtain faster shades. The substantive colors are also very useful in the dyeing of half-silk (silk and cotton fabrics), both for single colors and for the production of two-color effects, in the same manner as already described under their application to wool.

#### NOTES.

**1. The Substantive Colors on Wool.** — These dyes have gained considerable favor in various branches of wool dyeing, especially for knitting yarns fast to washing, carded wool and worsted yarns fast to milling, shoddy yarns, loose wool, and also for the dyeing of slubbing and yarns in machines. For the latter they are



especially adapted owing to their great solubility in water. The chief recommendation of these dyes to wool is their good fastness to washing, and in many cases excellent fastness to milling. This is especially true of the after-treated dyeings. Most of the substantive colors are also fast to stoving. The fastness of the substantive colors to washing is as a rule much better on wool than it is on cotton. The exhaustion of the bath is also generally better when wool is dyed. The after-treatment of the substantive colors on wool with chrome or chromium fluoride is usually carried out by adding the salts to the exhausted dye-bath, and using about one-half the weight of chrome as dyestuff or about the same weight of chromium fluoride as dyestuff. The dyeings may also be after-treated with bluestone in the same manner as with chrome, about the same weight of bluestone as dyestuff being taken. An after-treatment with both chrome and bluestone may be simultaneously effected in the same manner. In order to obtain a better exhaustion of the dye-bath for after-treatment it is best to add about 3 to 5 per cent. of acetic acid to the bath in finishing the dyeing; in case the bath does not exhaust well, it is best to carry out the after-treatment in a separate bath with the addition of 4 per cent. of acetic acid.

It should be observed that in dyeing wool with the substantive colors, neutral baths, or such as are but slightly acidulated with acetic acid, with few exceptions, are the most serviceable. If the baths are made too strongly acid, the color is taken up too rapidly by the wool, and uneven dyeings may result, which cannot afterwards be improved, even by prolonged boiling. In the case of material which is difficult to dye level, or if compound shades are not readily obtained, it is best to begin the dyeing without the addition of acid, and only when the greater part of the color has been taken up should the acid be added to the bath in order to increase the exhaustion. It should also be borne in mind that any vegetable matter present in the wool is not dyed so much in an acid bath as in a neutral one. Substantive dyes on wool after-treated with bluestone retain their remarkable fastness to light even after fulling, provided a neutral curd soap is used;

free alkali (which is generally present in soft soap) should never be present in the soap used under these circumstances.

**2. The Substantive Colors on Silk.** — As the majority of these colors may be dyed on silk, giving shades of considerable fastness to washing and water as well as to light, they are of considerable importance in this branch of dyeing. They are also useful for such goods that may be subjected to severe treatment with alkalis, such as fancy silk threads running through cotton or woollen fabrics. The substantive colors are best dyed on silk with the addition of Glaubersalt and acetic acid, or in a bath containing boiled-off liquor. If the dyeing is done without the latter, add to the bath for pale shades about 5 per cent. and for heavy shades about 10 per cent. of Glaubersalt, and only a small quantity (from 1 to 4 per cent.) of acetic acid at the beginning of the operation. If the color goes on the fibre too slowly, a little more acetic acid may be gradually added during the dyeing process. This precaution is necessary because it is difficult to obtain level colors if the bath is too acid at the beginning. For the same reason it is also important not to start the dyeing at too high a temperature. It is best to commence at 120° F. and slowly raise to the boil. For shading let the bath cool to 140° to 160° F., then add the necessary color solution and gradually heat up again. When dyeing light colors the baths exhaust, as a rule, with the addition of Glaubersalt only, or with a very little acetic acid added. For heavy shades the addition of 2 to 10 per cent. of acetic acid is necessary. When dyeing in a bath containing boiled-off liquor, for light shades the liquor need only be slightly acid, but for heavy shades the acidity should be greater. The brightening after dyeing may be done with either acetic or sulphuric acid, and the dyeings may be shaded in this bath in any desired manner. The fastness of the substantive colors on silk to acids, alkalies, stoving, and light corresponds in general to that which they possess when dyed on wool.

## SAMPLES.

- 279. Diamine Scarlet on wool; general method for dyeing.
- 280. Acid test on Diamine Scarlet.
- 281. Fulling test on Diamine Scarlet.
- 282. Showing use of ammonium acetate with Diamine Green.
- 283. Chrysophenine dyed in slightly acid bath.
- 284. Light test on Chrysophenine.
- 285. Union yarn dyed with Chrysophenine.
- 286. Union yarn dyed with Chrysophenine and Acid Violet.
- 287. Diamine Fast Red before treatment with chrome.
- 288. Diamine Fast Red after treatment with chrome.
- 289. Untreated dyeing tested to washing.
- 290. Chromed dyeing tested to washing.
- 291. Columbia Green before treatment with chromium fluoride.
- 292. Columbia Green after treatment with chromium fluoride.
- 293. Untreated dyeing tested to washing.
- 294. Treated dyeing tested to washing.
- 295. Showing Mikado Yellow does not dye wool.
- 296. Silk dyed with Benzo Fast Scarlet in soap bath.
- 297. Chrysophenine on silk; glauber salt and acetic acid bath.

## QUIZ 14.

- 416. What is the general method of dyeing wool with the substantive colors? Are these dyes used to any extent on wool? Do they yield fast colors?
- 417. What is the purpose of the addition of salt to the dye-bath with substantive colors on wool? What salts may be used?
- 418. Do the substantive dyes on wool show a tendency to uneven shades? Why?
- 419. How do the colors with the substantive dyes on wool compare with the acid and basic dyes as to brightness and depth of color?
- 420. How does Diamine Scarlet differ from Congo Red when tested with acid? Is this color fast to fulling? How is this test conducted?
- 421. For what purpose is ammonium acetate added to the bath when dyeing substantive colors on wool? Give the method of dyeing when using this salt.
- 422. What is probably formed when the ammonium acetate is boiled in the dye-bath?
- 423. How may the solution of ammonium acetate be prepared? How would you prepare one litre of a solution of ammonium acetate so that 10 cc. would contain 10 per cent. of the salt on 5 grams?
- 424. How would you prepare a solution of ammonium acetate so that one quart would be equivalent to 1 per cent. on 10 pounds of wool? So that one litre would be equivalent to 1 per cent. on 10 kilos?



425. Give the method of applying substantive colors to wool in a slightly acid bath. What acid should be employed and why?

426. What color does Chrysophenine give on wool, and what would you say as to its fastness to light?

427. What is meant by "union" material? What result is obtained when this is dyed with Chrysophenine? What kind of dye-bath is employed?

428. If union material is dyed in an acid bath with Chrysophenine and Acid Violet what will be the result?

429. What is the purpose of after-treating substantive dyes on wool with chrome? How is the process conducted?

430. How does the treatment with chrome affect the tone of Diamine Fast Red? Compare the tests to washing of the treated and untreated samples.

431. How is the after-treatment with chromium fluoride on substantive colors conducted? What influence does it have on the color? How does it differ in its action from chrome?

432. Compare the results of the tests to washing of the samples of Columbia Green treated with chromium fluoride and untreated.

433. Give an example of a substantive dye which does not color wool. What conditions are most favorable for a minimum amount of color on the wool? In what connection would such dyes be useful?

434. May the substantive dyes be satisfactorily applied to silk, and are the colors fast?

435. How are the substantive dyes applied to silk? Give two methods.

436. How may faster shades on silk with the substantive colors be obtained? What is meant by half-silk, and why are the substantive colors especially useful on such material?

437. On what character of wool material are the substantive dyes mostly used? Why are they so adaptable for dyeing in machines?

438. What is the chief recommendation of the substantive dyes on wool? Of what character of dyeings is this especially true? Are the substantive colors fast to stoving?

439. Does the fastness of the substantive colors differ when dyed on wool from that on cotton? How does the exhaustion of the dye-bath compare with the two fibres?

440. In general, how is the after-treatment of the substantive colors on wool with chromium salts conducted? How much chrome or chromium fluoride should be used?

441. How may the dyeings be after-treated with bluestone, and how much of this salt is taken? What is the principal effect of the bluestone on the fastness of the color?

442. What is the method of obtaining a better exhaustion of the dye-bath for after-treatment? In case the bath does not exhaust well, how should the after-treatment be conducted?



443. What is the most serviceable character of bath in dyeing wool with substantive colors? Why should the baths not be too strongly acid?

444. In case substantive dyes should show a tendency to uneven dyeing on wool, what precautions should be taken?

445. If vegetable matter is present in the wool when dyeing with substantive colors what character of bath should be used and why?

446. Are the substantive colors of importance in the dyeing of silk? Why are they serviceable for the dyeing of fancy silk threads?

447. How should light shades on silk be dyed with the substantive colors? Heavy shades?

448. What precautions should be taken in dyeing silk with substantive colors in order to obtain level shades?

449. How may silk be brightened after dyeing with substantive colors? Could Congo Red on silk be brightened, and why?

## SECTION XV.

### REPRESENTATIVE SUBSTANTIVE DYES ON COTTON.

**Experiment 98. Representative Substantive Dyes.** — Dye skeins of cotton yarn in baths containing 1 per cent. of soda ash, 20 per cent. of common-salt, and 2 per cent. of the respective dyes named below; enter at 160° F., bring to the boil, and dye at that temperature for one-half hour, then wash and dry. Use the following dyestuffs:

- (1) Thioflavine S (Cass.) (298).
- (2) Diamine Brown 3G (Cass.) (299).
- (3) Diamine Bordeaux B (Cass.) (300).
- (4) Diamine Orange D (Cass.) (301).
- (5) Chicago Blue 6B (Ber.) (302).
- (6) Columbia Black FB (Ber.) (303).
- (7) Diamine Rose BD (Cass.) (304).
- (8) Benzo Fast Scarlet (Elb.) (305).
- (9) Dianil Blue G (Metz) (306).
- (10) Dianil Green G (Metz) (307).

Test these dyeings for fastness to washing (308-317) and water (318-327).

Make a record of your results as follows:

Dyestuff.	Washing.			Water.	
	White wool.	White cotton.	Water.	White cotton.	Water.
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.....					
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## NOTES.

1. **Apparatus for Dyeing Cotton Yarn.** — Wooden vats are generally used for dyeing cotton in the form of hanks, and they are usually constructed to hold 50 or 100 pounds. It is seldom they are made larger or smaller than this for practical work. During the dyeing process the hanks are hung on smooth rods, so that only about one-fourth of their length is above the dye liquor. The yarn is turned by hand, or a stick may be used, in which case a pointed stick which is thinner than that on which the yarn hangs, is passed through the hank below the other stick, and the yarn is then raised with it and turned. The vats must be so constructed that the yarn can be easily turned without too much water being required in proportion to the cotton. The following are suitable internal dimensions:

For 50 pounds yarn	$\left\{ \begin{array}{l} \text{length, 64 inches.} \\ \text{breadth, } 22\frac{1}{2} \text{ inches.} \\ \text{depth, } 23\frac{1}{2} \text{ inches.} \end{array} \right.$
For 100 pounds yarn	$\left\{ \begin{array}{l} \text{length, 118 inches.} \\ \text{breadth, } 22\frac{1}{2} \text{ inches.} \\ \text{depth, } 23\frac{1}{2} \text{ inches.} \end{array} \right.$

The dye liquor is heated by a steam-coil which may enter the liquor at the top end of the vat. If the vats are long, two coils may be used, one from each end, but if short, one coil will be sufficient. These coils are closed at the ends, but the sides are suitably perforated with small holes, and it is best to fix them on to the steam pipe with a union joint so they may be removed from the vat if necessary. The steam-coil should lie under a perforated false bottom of wood, so as to prevent the yarn from coming in direct contact with the hot pipe, and also so that the force of the escaping steam may be broken and disseminated and not tangle up the yarn. In some cases, the pipe is fitted behind a perforated wooden partition which stands 4 to 6 inches from the top end of the vat, and which is a little lower than the latter. This arrangement offers certain advantages, as solutions of dyestuffs, etc.,

may be poured into the space behind it during the dyeing process and gradually distributed through the liquor without having to remove the yarn. To let the liquor run off after dyeing, it is best to have the vat fitted with a valve which can be opened by turning a handle from the outside. The old method of having a plug to be drawn is bad, as the workmen are liable to be scalded. The rods on which the yarn is hung should be hard straight sticks of hazel, ash, etc., from which all knots are removed so that no rough places are left. Besides the vat method of dyeing, cotton yarn may be dyed by machines; in one form a vat is used as with hand dyeing, but the sticks are turned mechanically by a system of interacting cogs. In another form, such as the Klauder-Weldon machine, the rods are arranged on a circular spider frame rotating in a semicircular vat, the sticks also being turned mechanically as the frame rotates; in this method, only one-half the load of yarn is in the liquor at any one time, so that economy in the amount of dye liquor is effected; the yarn is also kept out straight by being more or less stretched between the rods, which prevents tangling. Another method of machine dyeing, which has come into practice of late, and which may also be employed for yarns, though it is mostly used for cops, bobbins, etc., is where the material is tightly packed in a chamber of metal fixed to a suction tube and pump; the cotton remains stationary and the dyeing is effected by forcing the heated liquor through the material. Cotton yarn is also largely dyed in the form of prepared warps, in which case a special warp-dyeing machine is used, the warp or chain running over rollers up and down through the vat several times, then through squeeze rollers; if necessary several runs are made through the machine to obtain the desired shade. In the latter case, the machine may consist of several compartments each provided with squeeze rollers, and the yarn is run through each compartment successively.

**2. Apparatus for Dyeing Woolen Yarn.** — This material is usually dyed in wooden vats similar to those just described for the dyeing of cotton yarn. It must be borne in mind, however, that for woolen yarn dyed on sticks in large vats there will be



required about 300 to 450 gallons of water for 100 pounds of yarn, depending on the nature of the material. In general the yarn is entered into the hot acid dye liquor (in the case of acid dyes) and is dyed for  $1\frac{1}{2}$  to 2 hours at the boil. About 2 to  $3\frac{1}{2}$  pounds of yarn are placed on each stick, and for a 100-pound lot four men are generally employed in turning at the beginning, but later only two men are required. The yarn must not be turned more often than is necessary to obtain even colors, otherwise the fibres will become felted. From time to time the position of the sticks should be changed, that is, those in the middle of the vat should be moved to the ends, and so on. On a first kettle when dyeing woolen yarn it is usual to add a rather large quantity of Glaubersalt, often as much as 50 pounds on a 100-pound vat, this being a great help toward the production of level dyeings. On subsequent dyeings in the same vat only 5 pounds of Glaubersalt need be added. As a rule, old dye liquors cannot be used for longer than a week; though there are exceptional cases where they may be used continuously for several months. As a rule, the shades on woolen yarn are not perfectly level at the beginning of the process; but the color should distribute itself evenly after being boiled for some time; generally, the dyeings should be perfectly level a quarter of an hour after the color solution has been added to the bath. Woolen yarn may also be dyed in suitable machines similar to those used for cotton dyeing, the Klauder-Weldon form of machine being used extensively in this country, England, and Germany. This is especially true for fine worsted yarns, or in fact any kind of yarn which is easily matted or tangled; in such cases it is almost impossible to obtain satisfactory results by dyeing by hand in open vats on sticks, as the motion of the yarn and the boiling of the bath, especially if live steam is used for heating, cause a great felting and tangling of the threads. Such yarn is best dyed in a Klauder-Weldon machine, where the hanks may be stretched out and preserved in a straight condition throughout the dyeing operation, and no felting will result. Yarn which is liable to curl up, due to tight twist, should be scalded before washing or dyeing; this may be done by twisting

the hanks together tightly and laying them in boiling water for a couple of hours, then allowing them to cool before untwisting. In using the Klauder-Weldon machine for dyeing or scouring, this previous scalding will be superfluous, as the yarn will naturally undergo this operation when stretched in the machine during the dyeing process itself. With such yarn, however, the hanks should not be unstretched until they have passed through cold water or have cooled down.

**3. Apparatus for Dyeing Silk Yarn.** — Small lots of silk yarn are usually dyed in copper boilers, and larger lots in copper or copper-plated vats. These are usually mounted on wheeled frames, so that, with the exception of the long heavy vats, they may be conveniently moved about the dyehouse. This arrangement is a desirable one, as the dyer requires to use larger or smaller vats according to the quantity of silk which has to be dyed at one time. As the vats are often used for the most varying shades, it is necessary to frequently cleanse them thoroughly. For this purpose they may be first boiled out with old boiled-off liquor, or the inner sides may be thoroughly scoured with a hot strong solution of soda ash. After this has been run out, the vat is rinsed with water and then cleansed again with dilute sulphuric acid, and finally rinsed out again with water. The larger vats, which are stationary, are generally heated with a steam-coil placed under a perforated false bottom; for the smaller vats, usually movable steam-pipes are inserted. These steam-pipes should be fitted so as to turn in a ball-and-socket joint so that they may be moved around in any direction. The silk is hung in hanks on smooth rods in the same manner as wool or cotton, about one-half pound of silk being distributed on each stick. Silk may also be dyed in machines, the chief form in this country being the Klauder-Weldon, for which purpose a special machine is constructed. The spider is so arranged that at any time the entire lot of yarn may be raised out of the liquor.

**4. List of the Principal Substantive Dyes for Cotton.** — The substantive dyes form a very large and ever-increasing group. Although the most of them are applied almost exclusively to

cotton, nevertheless many of them are also used for dyeing wool and silk. Some of the substantive dyes are also adapted for after-treatment with bluestone and chrome. These are indicated in a separate list.

(a) RED.

Acetopurpurine 8B	Diamine Red B, 3B, 4B, 5B, 6B, 10B,
Alkali Bordeaux.	NO, and D.
Alkali Grenat.	Diamine Rose BD, BG, and GD.
Alkali Purpurine.	Diamine Scarlet B, 3B, and HS.
Alkali Red, and 7B.	Diamine Violet Red.
Azo Purpurine.	Dianil Ponceau G and 2R.
Benzo Bordeaux.	Direct Scarlet.
Benzo Fast Red L and GL.	Erika.
Benzo Fast Rose.	Geranine G and 2B.
Benzo Fast Scarlet.	Hessian Bordeaux.
Benzopurpurine B, 4B, 6B, and 10B.	Hessian Brilliant Purple.
Benzo Red 10B and SG.	Hessian Fast Rubine.
Benzo Rhoduline Red B and 3B.	Hessian Purple B, D, and N.
Brilliant Congo.	Oxamine Bordeaux.
Brilliant Geranine.	Oxamine Grenat.
Brilliant Purpurine 4B and 10B.	Oxamine Maroon.
Chicago Red.	Oxamine Red.
Chlorantine Red 4B and 8B.	Purpuramine.
Columbia Red 4B and 8B.	Rosazurine B and G.
Congo Red.	Rosophenine.
Congo Rubine.	Salmon Red.
Cotton Red 4B.	St. Denis Red.
Deltapurpurine 5B and 7B, and G.	Thiazine Red.
Diamine Bordeaux B and S.	Toluylene Red.
Diamine Brilliant Scarlet S.	Trona Red.
Diamine Fast Red F.	

(b) ORANGE.

Alkali Orange.	Dianil Orange.
Benzo Fast Orange S.	Direct Orange.
Benzo Orange R.	Mikado Orange.
Brilliant Orange G.	Naphthamine Orange.
Chicago Orange.	New Toluylene Orange.
Chloramine Orange.	Orange TA.
Columbia Orange.	Oxydiamine Orange.
Congo Orange G and R.	Pluto Orange G.
Cotton Orange G and R.	Pyramine Orange R and 3G.
Diamine Fast Orange.	Toluylene Orange.
Diamine Orange.	



## (c) YELLOW.

Alkali Yellow R.	Direct Yellow G and 3G, L, R, T, and TG.
Brilliant Yellow.	Hessian Yellow.
Carbazol Yellow.	Mekong Yellow.
Chloramine Yellow GG and M.	Mikado Gold Yellow.
Chlorantine Yellow T.	Mikado Yellow.
Chromine G.	Mimosa.
Chrysamine G and R.	Naphthamine Yellow.
Chrysophenine.	Oriol.
Columbia Yellow.	Oxydiamine Yellow.
Cotton Yellow G and R.	Oxydianil Yellow.
Curcumine S.	Polyphenyl Yellow.
Diamine Fast Yellow.	Salicine Yellow G and GG.
Diamine Gold.	Sun Yellow.
Diamine Yellow.	Thiazol Yellow G and R.
Dianil Yellow.	Thioflavine S.
Diphenyl Chrysoin.	Xanthine.
Diphenyl Citronine.	Yellow CR and D.
Diphenyl Fast Yellow.	

## (d) GREEN.

Alkali Green.	Diamine Dark Green.
Benzo Dark Green B and GG.	Diamine Green.
Benzo Green BB and G.	Direct Green.
Benzo Olive.	Eboli Green.
Brilliant Benzo Green.	Oxamine Dark Green.
Chloramine Green.	Oxamine Green.
Columbia Dark Green.	Tolamine Green.
Columbia Green.	

## (e) BLUE.

Acetylene Blue.	Benzo Indigo Blue.
Alkali Azo Blue.	Benzo Marine Blue.
Azo Blue.	Benzo Pure Blue.
Azo Corinth.	Benzo Red Blue G.
Azo Dark Blue.	Brilliant Azurine.
Azo Mauve.	Brilliant Benzo Blue 6B.
Benzo Azurine G, 3G, and R.	Chicago Blue.
Benzo Blue.	Chlorazol Blue.
Benzo Chrome Dark Blue B and N.	Columbia Blue.
Benzo Copper Blue B.	Columbia Fast Blue.
Benzo Cyanine B, 3B, and R.	Columbia Dark Blue.
Benzo Dark Blue R, G, and 5G.	Congo Blue.
Benzo Fast Blue.	Congo Fast Blue.



(e) BLUE. — Continued.

Congo Pure Blue.	Erie Blue BX and 2G.
Diamine Azo Blue R and 2R.	Naphthamine Blue.
Diamine Blue BX, RW, BG, 2B, 3B.	Naphthamine Deep Blue.
Diamine Brilliant Blue G.	Naphthamine Indigo.
Diamine Dark Blue.	Naphthazurine.
Diamine Fast Blue.	Naphthyl Blue BB.
Diamine New Blue.	New Toluylene Blue.
Diamine Pure Blue.	Oxamine Blue.
Diamine Sky Blue.	Oxamine Dark Blue.
Diamine Steel Blue.	Oxydiamine Blue R, 3R, and G.
Diamineral Blue.	Phenamine Blue.
Diphenyl Blue 3G.	Toledo Blue V.
Direct Blue B, 2BX, and 3BX.	Toluylene Blue.
Direct Blue B and R.	Toluylene Dark Blue.
Direct Indigo Blue.	Triamine Blue.
Eboli Blue B, 6B, and 2R.	Triazol Blue.

(f) VIOLET.

Alkali Azo Violet.	Congo Violet.
Azo Corinth.	Diamine Heliotrope G, O, and B.
Azo Gallein.	Diamine Violet N.
Azo Mauve.	Dianil Bordeaux.
Azo Violet.	Direct Violet.
Benzo Fast Violet.	Heliotrope.
Benzo Violet.	Hessian Bordeaux.
Bordeaux Extra.	Hessian Violet.
Bordeaux COV.	Oxamine Violet.
Chloramine Violet.	Oxydiamine Violet B, R, and G.
Chlorantine Lilac.	Triazol Violet.
Clemantine.	Trisulphon Violet.
Congo Corinth.	

(g) BROWN.

Alkali Brown.	Chlorantine Brown.
Alkali Dark Brown.	Columbia Brown.
Alkali Red Brown.	Congo Brown.
Benzo Brown.	Cotton Brown.
Benzo Chrome Brown.	Diamine Bronze.
Catechu Brown.	Diamine Brown.
Chicago Brown.	Diamine Catechine.

## (g) BROWN. — Continued.

Diamine Fast Brown.  
 Diamine Nitrazol Brown.  
 Diamineral Brown.  
 Diazo Brown.  
 Diphenyl Brown.  
 Direct Brown.  
 Direct Bronze Brown.  
 Direct Fast Brown.  
 Fast Cotton Brown.  
 Hessian Brown.  
 Mikado Brown.

Naphthamine Brown.  
 New Toluylene Brown.  
 Oxamine Brown.  
 Oxamine Maroon.  
 Oxydiamine Brown.  
 Pluto Brown.  
 Terra Cotta.  
 Thiazine Brown.  
 Toluylene Brown.  
 Trisulphon Brown.  
 Zambesi Brown.

## (h) BLACK.

Alkali Black B and G.  
 Benzo Chrome Black.  
 Benzo Chrome Blue Black.  
 Benzo Fast Black.  
 Carbide Black.  
 Chromanil Black.  
 Cold Black.  
 Columbia Black.  
 Cotton Black.  
 Diamine Black.  
 Diamine Blue Black.  
 Diamine Deep Black.  
 Diamine Jet Black.  
 Diamineral Black.  
 Diaminogene B, and extra.  
 Dianil Black.  
 Diazo Black.  
 Diazo Blue Black.

Diphenyl Blue Black.  
 Diphenyl Black.  
 Diphenyl Fast Black.  
 Direct Black.  
 Direct Blue Black.  
 Direct Deep Black.  
 Isodiphenyl Black.  
 Melantherine.  
 Naphthamine Black.  
 Nyanza Black.  
 Oxamine Black.  
 Oxydiamine Black.  
 Pluto Black.  
 Polyphenyl Black.  
 Tabora Black.  
 Toluylene Black.  
 Violet Black.  
 Zambesi Black.

## (i) GRAY.

Benzo Fast Gray.  
 Chicago Gray.  
 Diamine Gray.  
 Diphenyl Gray.  
 Direct Gray.

Fast Gray.  
 Hessian Copper Gray.  
 Hessian Gray.  
 Neutral Gray.  
 Zambesi Gray.

### 5. Substantive Dyes Suitable for After-treatment with Blue-stone.

Azo Violet.	Brilliant Benzo Blue 6B.
Benzo Azurine G, 3G, and R.	Chloramine Violet R.
Benzo Blue.	Chrysamine G and R.
Benzo Copper Blue B.	Diamine Blue RW.
Benzo Cyanine B, 3B, and R.	Diamine Pure Blue.
Benzo Pure Blue.	Hessian Copper Gray.
Brilliant Azurine.	Pluto Orange G.

### 6. Substantive Dyes Suitable for After-treatment with Chrome and Bluestone.

Benzo Chrome Black B and N.	Columbia Chrome Black.
Benzo Chrome Blue Black B.	Cupranil Brown.
Benzo Chrome Brown.	Diamine Catechine.
Carbide Black BO.	Diamineral Black.
Catechu Brown.	Direct Deep Black E extra.
Chromanil Black.	Direct Deep Black RW extra.
Chromanil Brown.	Pluto Orange G.
Chrysamine G and R.	Trisulphon Brown S.

### 7. Principal Substantive Dyes Applicable to Wool.

#### (a) RED.

Anthracene Red.	Delta Purpurine 5B and 7B.
Benzo Bordeaux 6B.	Diamine Bordeaux B and S.
Benzo Fast Red L.	Diamine Fast Red F.
Benzo Purpurine B, 4B, 6B, and 10B.	Diamine Red NO, D, 5B, and 10B.
Benzo Red 10B.	Diamine Scarlet B, 3B, and HS.
Benzo Rhoduline Red.	Geranine G and BB.
Brilliant Congo R.	Hessian Bordeaux.
Brilliant Geranine B.	Hessian Brilliant Purple.
Brilliant Purpurine R.	Hessian Purple.
Congo Red.	Rosazurine B, G.
Congo Rubine.	

#### (b) ORANGE.

Alkali Orange.	Direct Orange R and 2R.
Benzo Fast Orange S.	Mikado Orange.
Brilliant Orange G.	Orange TA.
Congo Orange G and R.	Pluto Orange G.
Cotton Orange G and R.	Toluylene Orange G and R.
Diamine Orange B, G, D, GC, and DC.	

## (c) YELLOW.

Brilliant Yellow.  
 Carbazol Yellow.  
 Chloramine Yellow M.  
 Chrysamine G and R.  
 Chrysophenine.  
 Cotton Yellow R.  
 Curcumine S and W.  
 Diamine Fast Yellow A.  
 Diamine Gold Yellow.  
 Diamine Yellow N.

Diphenyl Citronine.  
 Diphenyl Fast Yellow.  
 Hessian Yellow.  
 Mikado Yellow.  
 Polyphenyl Yellow.  
 Salicine Yellow G, 2G.  
 Sun Yellow 3G.  
 Thiazol Yellow G and R.  
 Thioflavine S.

## (d) GREEN.

Benzo Dark Green B, 2G.  
 Benzo Green 2B and G.

Diamine Green B.  
 Eboli Green S and ST.

## (e) BLUE.

Azo Blue.  
 Benzo Azurine G, 3G, and R.  
 Benzo Blue 2B, 3B, and BX, 2R, 4R,  
 and RW.  
 Benzo Cyanine B and R.  
 Chicago Blue B.  
 Congo Blue 2B.  
 Diamine Blue 2B, 3B, and BX, 3R,  
 and RW.  
 Diamine Brilliant Blue.

Diamine New Blue G and R.  
 Diamine Pure Blue FF.  
 Eboli Blue.  
 Toledo Blue.

Most of the blue substantive dyes exhaust but poorly on wool from a neutral bath; better results are obtained by the addition of a small amount of acetic acid, or under some conditions, of sulphuric acid.

## (f) VIOLET.

Azo Violet.  
 Chloramine Violet.  
 Congo Corinth B and G.

Diamine Violet N.  
 Diazo Black H and R.  
 Hessian Violet.

## (g) BROWN.

Benzo Brown.  
 Benzo Chrome Brown G, 5G, R, and  
 3R.  
 Cotton Brown A and N.  
 Diamine Bronze G.  
 Diamine Brown B, 3G, M, and V.

Diazo Brown G.  
 Direct Fast Brown B and 2G.  
 Pegu Brown.  
 Pluto Brown 2G, NB, and R.  
 Toluylene Brown.



(h) BLACK.

Benzo Fast Black.	Diazo Black 3B, BHN.
Diamine Black BH, HW.	Direct Deep Black E extra, and RW.
Diamine Deep Black 2S, 2O.	Oxydiamine Black N, SOOO.

(i) The following colors may also be applied to wool if a considerable amount of acetic acid be added to the dye-bath.

Benzo Fast Scarlet 4BS and GS.	Brilliant Benzo Blue 6B.
Benzo Fast Violet R.	Chloramine Orange G.
Benzo Orange R.	Direct Blue Black B, 2B, and N.
Benzo Pure Blue 4B.	Direct Deep Black G.
Benzo Violet R.	Heliotrope 2B.
Brilliant Azurine B, G, and 5G.	Pluto Black G and F.

SAMPLES.

- 298. Thioflavine S on cotton.
- 299. Diamine Brown 3G on cotton.
- 300. Diamine Bordeaux B on cotton.
- 301. Diamine Orange D on cotton.
- 302. Chicago Blue 6B on cotton.
- 303. Columbia Black FB on cotton.
- 304. Diamine Rose BD on cotton.
- 305. Benzo Fast Scarlet on cotton.
- 306. Dianil Blue G on cotton.
- 307. Dianil Green G on cotton.
- 308-317. Tests for fastness to washing.
- 318-327. Tests for fastness to water.

QUIZ 15.

450. What color does Thioflavine S give on cotton? Name five substantive dyestuffs and the firms which manufacture them.

451. Of the ten substantive dyestuffs tested, which ones were found to be the fastest to washing? How was the washing test made?

452. Which of the substantive dyes were found to be fastest to water? How was the water test made?

453. Describe the necessary apparatus required for dyeing 100 pounds of cotton by hand. What is the usual breadth and depth of dye-vats?

454. What is the usual length of a vat for dyeing 50 pounds of cotton yarn? For dyeing 100 pounds?

455. How may the dye liquor be heated in the vat? Why should a perforated false bottom be used? What advantage is there in having the coil in an end partition?

456. What character of rods should be used for hanging the hanks of yarn on?

457. In what forms of machines may cotton yarn be dyed? Describe the Klauder-Weldon machine. What advantages does this offer?

458. Describe the general principle of the pressure or suction dyeing machines. What advantages and disadvantages do machines of this type possess?

459. How are cotton warps dyed? What are the advantages of warp dyeing?

460. Assuming a breadth of 22 inches and a depth of 24 inches, a vat of what length would be required for the dyeing of 100 pounds of woolen yarn, supposing 600 gallons U. S. to be used in the dye-vat?

461. How much woolen yarn may be placed on each stick in the dye-vat, and how many men are required for turning? Supposing that 4 men at \$1.25 per day of 10 hours can run two vats of 100 pounds each, and that the time of dyeing, washing, etc., for each lot is  $2\frac{1}{2}$  hours, what would be the wage cost of dyeing 2000 pounds of woolen yarn?

462. In using standing kettles in dyeing 100-pound lots, with how much glaupersalt is the first bath charged generally in practice? How much is added to subsequent baths?

463. For what length of time, as a rule, may standing kettles be run when dyeing woolen yarn in acid baths?

464. Does the color on woolen yarn, as a rule, come up level on first starting the dyeing? What reason do you assign to this? What causes the color to distribute itself evenly and in what time should it level up?

465. What character of machines may be used for dyeing woolen yarn? For what kind of yarn is the Klauder-Weldon machine especially adapted?

466. Woolen yarn which is liable to curl up should be treated in what manner before dyeing? Give an explanation of this process.

467. Is a previous scalding of curly yarn necessary when using a Klauder-Weldon machine for dyeing? Why?

468. What kind of vats are used for the dyeing of silk yarns? Explain the manner of cleaning the vats when using different colors.

469. How much silk should be placed to a stick in dyeing by hand? In what character of machines may silk yarn be dyed?

470. Describe the Klauder-Weldon machine employed especially for the dyeing of silk.

## SECTION XVI.

### APPLICATION OF MORDANT DYES TO WOOL.

**Experiment 99. General Method of Dyeing.** — The most generally used mordant for wool is chrome or potassium bichromate. It is applied to the fibre in the following manner: Prepare a bath containing 3 per cent. of chrome and 4 per cent. of tartar; enter a test-skein of woolen yarn at  $140^{\circ}$  F.; gradually raise to the boil, and continue at that temperature for one-half hour; wash well, and then dye in a fresh bath containing 2 per cent. Alizarin Blue NG and 4 per cent. of calcium acetate; enter at  $100^{\circ}$  F., gradually raise to the boil, and dye at that temperature for one-half hour; then add 2 per cent. of acetic acid and boil for 15 minutes longer; wash well and dry (328). Chrome, or potassium bichromate, is a salt of chromic acid ( $\text{CrO}_3$ ), while the mordant which is eventually produced on the fibre is chromium oxide ( $\text{Cr}_2\text{O}_3$ ); hence in the process of mordanting the chrome must undergo reduction. This is brought about partly by the wool itself, but chiefly by the aid of the tartar. The latter is potassium acid tartrate, or potassium bitartrate, and is a reducing agent. After mordanting it will be noticed that the wool is yellow in color; this is probably due to the formation of chromium chromate in the fibre. If this compound is exposed to the action of strong light it will suffer a rapid reduction to chromium oxide, which is green in color; hence it is best not to expose the mordanted wool unevenly to light for any length of time before dyeing.

**Experiment 100. Effect of Iron Salts in the Bath.** — Alizarin colors are much affected by the presence of iron salts in either the mordant or the dye-bath, the color being considerably dulled through the formation of an iron color-lake with the dyestuff.



To show this influence in the mordant bath, mordant a skein of woolen yarn in a bath containing 3 per cent. of chrome, 4 per cent. of tartar, and a few drops of a solution of copperas. After mordanting (329), dye as usual with 2 per cent. of Alizarin Blue NG; wash and dry (330), and compare the color thus obtained with that produced with the same mordant and dyestuff without the addition of the iron salt. Mordant a second skein of woolen yarn in the usual manner with 3 per cent. of chrome and 4 per cent. of tartar, and dye as before with 2 per cent. of Alizarin Blue NG, but add to the dye-bath a few drops of a solution of copperas. Notice the effect of this on the appearance of the color (331).

**Experiment 101. Comparison of Different Mordants on Wool.**—Mordant a skein of woolen yarn in a bath containing 3 per cent. of chrome and 4 per cent. of tartar; enter at 140° F., gradually bring to the boil, and continue at that temperature for one-half hour; wash well (332) and dye in a fresh bath containing 2 per cent. of Alizarin Red SW; enter at 100° F., gradually bring to the boil, and dye at that temperature for one-half hour, then wash well and dry (333).

Mordant a second skein of woolen yarn in the same manner in a similar bath, but instead of using chrome use 10 per cent. of alum; wash well (334), and dye as before with 2 per cent. of Alizarin Red SW (335).

Mordant a third skein, using 8 per cent. of copperas as the mordant with 6 per cent. of tartar; wash well (336), and dye with 2 per cent. of Alizarin Red SW (337).

Mordant a fourth skein with 5 per cent. of bluestone (338) and 4 per cent. of tartar; wash well, and dye with 2 per cent. of Alizarin Red SW (339).

Mordant a fifth skein with 4 per cent. of stannous chloride and 2 per cent. of oxalic acid; wash well (340), and dye with 2 per cent. of Alizarin Red SW (341).

Compare the several colors obtained on the different mordants with the same dyestuff, and also preserve samples of the original mordanted yarn before dyeing in each case, so as to be able to



compare the colors given by the mordants alone. Make a record of your results as follows:

Mordant.	Color of mordanted skein.	Color of dyed skein.
Chromium .....	.....	.....
Aluminium .....	.....	.....
Iron.....	.....	.....
Copper.....	.....	.....
Tin.....	.....	.....

**Experiment 102. After-Mordanting with Chrome.** — This method may be used with quite a number of the alizarin colors, and is becoming a favorite process, as only one bath is required. Dye a skein of woolen yarn in a bath containing 2 per cent. of Anthracene Yellow C, 2 per cent. of sulphuric acid, and 20 per cent. of Glaubersalt; enter at 120° F., gradually bring to the boil, and continue at that temperature for one-half hour; then lift the skein (342a) and add 3 per cent. of chrome, and continue the boiling for 15 minutes; wash and dry (342b). Preserve a sample of the color before chroming and compare it with the chromed color. Many of the alizarins are now prepared in such a manner that they have slight acid properties and are capable of being absorbed by the wool fibre from acid baths; these alizarins are chiefly in the powder form, and are compounds of the dyestuffs with sodium bisulphite; they are also much more soluble in water than the ordinary alizarins.

Dye a skein of woolen yarn in a bath containing 4 per cent. of acetic acid, 2 per cent. of Acid Alizarin Green R, and 20 per cent. of Glaubersalt (343a). Dye in the same manner as before and then add 3 per cent. of chrome as above; wash well and dry (343b).

Dye a third skein in a bath containing 2 per cent. of sulphuric acid, 4 per cent. of Diamond Black GA, and 20 per cent. of Glaubersalt (344); dye as before and after-chrome with 2 per cent. of chrome and 1 per cent. of sulphuric acid; wash well and dry (345).

Dye a fourth skein with 2 per cent. of Diamond Flavine (346) in the same manner and after-chrome; wash and dry (347). In each case preserve a sample of the skein before chroming in order to observe any change in the color due to the chroming.

Test these colors as to their fastness to washing and acids.

Make a record of your results in the following manner:

Dyestuff.	Effect of chroming.	Washing test.			Acid test.
		Color.	White wool.	White cotton.	
Anthracene Yellow C .....					
Acid Alizarin Green R .....					
Diamond Black GA. ....					
Diamond Flavine.....					

#### NOTES.

1. **The Mordanting of Wool.** — In mordanting wool with chrome the mordanting bath is not exhausted and may be used again for fresh lots of wool, adding about 2 per cent. of chrome and 3 per cent. of tartar each time. After mordanting the wool should be well washed in order to remove the excess of mordanting liquor from the fibre, which coming in contact with the dye liquor would cause a loss of coloring-matter by precipitation and also form a loosely adherent surface color-lake on the fibre which would eventually rub off badly and cause the color to smut or crock. It has been demonstrated that about 3 per cent. of chrome is the proper amount of mordant to employ for full shades of alizarin colors; for lighter shades less mordant may be used. If larger quantities than 3 per cent. are employed, the color is liable to be injured and will not be as heavy or as bright as when only 3 per cent. is used. The use of too much chrome also has the effect of oxidizing the wool fibre itself, causing it to become harsh, and with some dyes to take up less coloring-matter. In place of using tartar as the assistant in the mordanting process,

there may be employed such substances as oxalic acid, lactic acid, formic acid, lactoline (which is potassium bilactate), liginosin (which is a substance obtained from the spent liquors in the bisulphite method of bleaching wood pulp); sulphuric or hydrochloric acids may also be used, and sodium bisulphate (which is sold as "tartar substitute") is frequently employed where cheapness is more desirable than quality. It is the general opinion, however, that tartar furnishes the best all-round results. Lactic acid employed in connection with sulphuric acid is a very good assistant where heavy shades, such as blues, browns, etc., are to be dyed; it causes a complete reduction and exhaustion of the mordanting bath and only requires the use of about 2 per cent. of chrome in place of the usual 3 per cent.; it causes the mordanted wool to have a very decided greenish color, however, and on this account does not give as good results as tartar in certain shades. The use of formic acid as a chrome assistant is becoming of some importance.

**2. Dyeing Wool with Mordant Colors.** — Calcium acetate is added to the dye-bath for the purpose of brightening the color, and it is supposed that a triple color-lake is formed between the chromium, the calcium, and the alizarin. Where the water employed for the dye-bath is sufficiently hard (that is, contains sufficient lime salts in solution) the addition of acetic acid in requisite amounts will form the necessary calcium acetate, hence none of this salt need be added under such conditions. For water of 5 degrees to 10 degrees of hardness (one degree of hardness represents 1 part of lime in 100,000 parts of water) 2 parts of acetic acid (of 9° Bé.) should be added for each 1000 parts of water in the dye-bath; and for water of 10 degrees to 15 degrees hardness 3 parts of acetic acid should be added. The best and most practical way, perhaps, is to add acetic acid to the dye-bath until a test-paper of blue litmus is distinctly reddened. Acetic acid is furthermore added to the dye-bath for the purpose of more thoroughly exhausting the coloring-matter, but the addition of the acid in this case should not be made until near the end of the dyeing operation, in order to prevent unevenness.



The alizarins, as a rule, exhaust quite well and many of them will not require any acid at all, especially when light shades are dyed. The initial temperature of the alizarin dye-bath should be quite low (100° F. or even lower), and the elevation of the temperature to the boil should be gradual in order to have the dyeing even and well penetrated. The color-lake does not develop fully until after boiling for some time, hence it requires a longer time, as a rule, to dye alizarins than it does acid colors on wool. In dyeing the after-mordanted alizarins, it is usually the plan to add the mordant to the dye-bath after the dyeing is completed; for this purpose, the dye-bath should be very well exhausted, otherwise the mordanting would require a fresh bath. At times, where light shades are being dyed, the mordant and the dyestuff (both being in small quantities) may be added to the bath at once, and the mordanting and dyeing take place simultaneously. There is a preparation known as "Metachrome" mordant which consists essentially of chrome and ammonium sulphate, which is used together with certain alizarins for dyeing in a single bath. The presence of the ammonium sulphate prevents the formation of the color-lake before the dye-bath has reached the boil, and by that time most of the chrome will have been absorbed by the fibre. This process with some modifications has lately become of considerable practical value for the dyeing of worsted yarns, ammonium acetate being used in conjunction with chrome in the dye-bath. Only certain of the mordant colors are applicable; these are sold as "chromate" dyes, etc. Where very bright colors are desired, as with reds, blues, and yellows, chrome cannot be used as the mordant, but alum or stannous chloride may be employed. Alum is used to quite an extent for certain shades, but as the colors obtained on a tin mordant are not as fast and as the tin mordant makes the wool harsh and brittle, it is very little used in practice. In order to obtain as bright and clear colors as possible with the alizarin dyes, it is necessary that the water and the chemicals employed both for mordanting and dyeing should be free from any trace of iron, as the presence of this metal causes a saddening of the color.



**3. List of the Principal Mordant Dyes.****(a) APPLICABLE TO PREVIOUSLY MORDANTED WOOL.**

Alizarin.	Chromazurine S
Alizarin Black B, R, 2R, and V.	Chrome Blue.
Alizarin Blue (all brands).	Chrome Brown.
Alizarin Blue Black W and SW.	Chromocyanine B and V.
Alizarin Blue S (all brands).	Cloth Brown.
Alizarin Bordeaux.	Cœlestine Blue B.
Alizarin Brown (all brands).	Cœruleine.
Alizarin Chrome Black W.	Cœruleine S in paste.
Alizarin Cyanine (all brands).	Coreine 2R, AB, and AR.
Alizarin Dark Blue SW, and 2W, and S.	Delphine Blue.
Alizarin Gray G and R.	Diamond Brown paste.
Alizarin Green SW and S.	Dioxine.
Alizarin Indigo SW and SMW.	Gallamine Blue.
Alizarin Maroon.	Gallanil Indigo PS
Alizarin Reds (pastes).	Gallocyanine.
Alizarin Viridine FF and DG.	Galloflavine.
Alizarin Yellow FS.	Gallozine A.
Alizarin Yellow N powder.	Gambine G and R.
Anthracene Blue.	Phenocyanine B and VS.
Anthracene Brown (all brands).	Prune.
Anthracene Dark Blue W.	Resoflavine.
Anthracyl Blue G and R.	Rufigallol.
Azo Chromine G.	Sulphamine.
Blue PRC.	Sulphamine Brown A and B.

**(b) SUITABLE FOR AFTER-MORDANTING.**

Acid Alizarin Black.	Chrome Black B and T.
Acid Alizarin Blue BB and GR.	Chrome Fast Black B.
Acid Alizarin Brown B.	Chrome Fast Black F, R, and BB.
Acid Alizarin Gray.	Chrome Patent Black.
Acid Alizarin Green.	Diamond Black (all brands).
Acid Alizarin Grenat.	Diamond Brown 3R.
Acid Alizarin Yellow.	Diamond Green B.
Acid Anthracene Brown R, T, and W.	Domingo Chrome Black.
Acid Chrome Black B and G.	Domingo Violet Black.
Alizarin Cyanine Green.	Palatine Chrome Black A.
Anthracene Acid Brown (all brands).	Palatine Chrome Brown W.
Anthracene Chrome Black.	

## (c) SUITABLE FOR BOTH METHODS OF MORDANTING.

Alizarin Black (all brands).	Cloth Red (all brands).
Alizarin Blue Black B and 3B.	Cloth Scarlet.
Alizarin Cyanine Black G.	Diamond Flavine G.
Alizarin Fast Black T.	Diamond Orange paste.
Alizarin Orange paste (all brands).	Diamond Yellow G paste.
Alizarin Red PS powder.	Domingo Chrome Red.
Alizarin Red S powder.	Domingo Chrome Yellow.
Alizarin Red SB and W.	Fast Brown.
Alizarin Red WS.	Fast Mordant Yellow.
Alizarin Yellow A, C paste.	Gallanil Violet.
Alizarin Yellow paste.	Galleine.
Alizarin Yellow 2GW, R, RW, 2G, 3G, and R.	Indochromine.
Anthracene Red.	Metachrome Brown B.
Anthracene Yellow (all brands).	Milling Brown B and G.
Anthracyl Fast Red.	Milling Orange.
Brilliant Alizarin Blue G and R.	Milling Red.
Brilliant Alizarin Cyanine G and 3G.	Milling Yellow.
Carbazol Yellow W.	Mordant Yellow G, R, and O.
Chrome Fast Yellow G.	Salicine Red.
Chrome Patent Green.	Salicine Yellow G and 2G.
Chrome Yellow (all brands).	Wool Red.
Cloth Orange.	Wool Yellow.

## (d) SUITABLE FOR DYEING IN AN ACID BATH WITHOUT AFTER-CHROMING.

Alizarin Cyanine Green.	Diamond Brown 3R.
Alizarin Heliotrope.	Fast Green G.
Alizarin Irisol.	Milling Green S.
Alizarin Pure Blue.	Naphthol Green B.
Alizarin Saphirlol.	

## (e) CHROME DEVELOPED DYESTUFFS.

Acid Alizarin Black R.	Chrome Brown BO and RO.
Azo Fuchsine B and G.	Chromogen I.
Azo Rubine.	Chromotrop (all brands).
Carmoisine B	Florida Red.

**SAMPLES.**

- 328. Alizarin Blue on wool; showing general method of dyeing.
- 329. Effect of iron in mordant bath.
- 330. Effect of iron mordant on color.
- 331. Effect of iron in dye-bath.
- 332. Wool mordanted with chrome.
- 333. Alizarin Red on a chrome mordant.
- 334. Wool mordanted with alum.
- 335. Alizarin Red on alum mordant.
- 336. Wool mordanted with copperas.
- 337. Alizarin Red on a copperas mordant.
- 338. Wool mordanted with bluestone.
- 339. Alizarin Red on a bluestone mordant.
- 340. Wool mordanted with stannous chloride.
- 341. Alizarin Red on a tin mordant.
- 342a. Anthracene Yellow on wool before chroming.
- 342b. Anthracene Yellow after chroming.
- 343a. Acid Alizarin Green before chroming.
- 343b. Acid Alizarin Green after chroming.
- 344. Diamond Black GA before chroming.
- 345. Diamond Black GA after chroming.
- 346. Diamond Yellow before chroming.
- 347. Diamond Yellow after chroming.

**QUIZ 18.**

- 471. What is the most generally used mordant on wool? How is it applied to the fibre?
- 472. Give the general method for dyeing the alizarin colors on a chrome mordant.
- 473. What is chrome? Of what acid is it a salt? In what condition is the mordant eventually produced on the fibre?
- 474. How is the reduction of the chrome brought about in the mordanting bath?
- 475. What is tartar? How does it act as an assistant in mordanting with chrome?
- 476. How do iron salts in the mordant affect the colors of alizarin dyes? Should iron be present in the dye-bath?
- 477. What are the five principal mordants which may be used on wool? Of what metals are they derivatives?
- 478. What color does a chrome mordant give on wool? Why should not chrome mordanted wool be exposed for any length of time to strong light before dyeing?

479. What color does Alizarin Red give with a chrome mordant? With a copperas mordant?

480. What is copperas? How is this mordant applied to wool? In what condition would the mordant be present in the fibre? What color does the mordant give to the wool?

481. What is bluestone? How is it applied to wool as a mordant? What color does it give to the fibre?

482. What color does Alizarin Red give with a copper mordant? Compare this color with that obtained with chrome.

483. What is the tin mordant used on wool? How is it applied? What color does it give to the fibre? How does it affect the physical properties of the fibre?

484. What color does Alizarin Red give with a tin mordant? How does this color compare with that obtained with chrome?

485. How is the process of after-mordanting carried out? How many baths are required?

486. What effect does the after-chroming process have on the color of anthracene yellow?

487. What kind of alizarin dyes are best adapted for the after-chroming process? In what form do such dyes usually come, and of what do they consist? How do they compare with ordinary alizarins as to solubility?

488. How does the after-chroming affect the color of Acid Alizarin Green?

489. Describe the method of dyeing Diamond Black. What are the characteristics of this color?

490. Give the process of dyeing Diamond Yellow. What is the fastness of this color to washing and acids?

491. In using a chrome mordant bath as a standing kettle how much chrome and tartar must be added for successive lots?

492. Why should wool be well washed after mordanting? What would be the result if this were not done?

493. What is the proper amount of chrome to use in preparing a mordant bath for a full shade? Is this amount necessary for lighter shades?

494. Why is it not desirable to employ larger amounts than 3 per cent. of chrome in mordanting wool? What is the effect of too much chrome on the fibre itself?

495. What other substances besides tartar may be used as assistants in the mordant bath? What are lactoline, lignorosin, and tartar substitute?

496. Which assistant furnishes the best results? For what colors is lactic acid a good assistant? What is the advantage of lactic acid as compared with tartar? What color does the wool acquire with lactic acid, and why?

497. Why is calcium acetate often added to the dye-bath with alizarin colors? Why is hard water beneficial when dyeing alizarin colors?



498. In using hard water with alizarin colors why is acetic acid used in the dye-bath? What proportion of acetic acid should be added to water of 10 degrees hardness? What is meant by a degree of hardness? What is the most practical method of knowing the proper amount of acetic acid to use in the bath?

499. What further use has acetic acid in dyeing alizarin colors besides the formation of calcium acetate with the lime salts in the water?

500. Do the alizarin dyes exhaust well? How may the degree of exhaustion be increased? How is unevenness prevented?

501. Why should the alizarin dye-bath be started at a low temperature, and why is a good boiling necessary?

502. Under what conditions may the mordant and dyestuff be added together to the dye-bath? What is metachrome mordant and how is it used?

503. When especially bright colors are to be obtained with alizarins what mordants must be employed? What precautions should be taken in order to obtain clear bright colors?

504. What is meant by saddening a color, and how may this be done in the case of alizarin dyes?

## SECTION XVII.

### DEVELOPED DYES ON COTTON AND SILK.

**Experiment 103. General Method of Applying Developed Dyes.** — Certain of the substantive dyes may be applied to cotton in the usual manner, and then changed by chemical treatment into other dyestuffs which may be of a totally different color, and are frequently much faster or deeper in shade than the original color from which they have been derived. In other words, the dyestuff is built up within the fibre itself just as ordinary dyestuffs are formed without reference to the fibre. This class of substantive dyes is known as the "developed" or "diazotized" colors, from the chemical processes through which they pass. These dyes form a rather important class of colors, the value and adaptability of which are constantly growing. Primuline was the first of these dyes discovered, and is still the most important one in use and may be taken as the type of the entire class. Dye a test-skein of cotton yarn in a bath containing 6 per cent. of Primuline, 20 per cent. of salt, and 1 per cent. of soda ash; enter at 140° F., gradually raise to the boil and dye at that temperature for one-half hour. It will be noticed that this is simply the general method for applying substantive dyes, and that the color obtained is yellow (348). Rinse the skein in fresh water and pass into a cold bath containing 5 per cent. of sodium nitrite and 6 per cent. of sulphuric acid; work for about 10 minutes. It will be noted that the yellow color of the dye is altered to a brownish yellow (349) by this treatment, and if the odor of the bath is observed the presence of nitrous acid will be noted. Rinse the skein with cold water, and immediately pass into a third bath containing 2 per cent. of beta-naphthol solution; work cold for 15 minutes, then wash well and dry (350). When placed in the third bath it will be noticed that the skein turns a bright red color, which is due

to the new dyestuff which has thus been formed within the fibre. In the first bath the Primuline acts merely as a substantive dye, and gives a yellow color which possesses no fastness and is unimportant. The second solution is termed the "diazotizing" bath. The action of the sodium nitrite on the sulphuric acid is to liberate nitrous acid and form sodium sulphate; the nitrous acid acts on the dyestuff in such a manner that the amido groups ( $\text{NH}_2$ ) present are changed into what are known as "diazo" groups,  $\text{N} : \text{N}$ . This diazo group combines with the sulphuric acid present in the bath and forms primuline-diazo-sulphate. The diazo body is quite unstable, hence the bath must be employed cold, and the cotton must be passed from this bath as soon as possible into the third bath, for if the diazotized material is allowed to stand for any length of time, especially if exposed to strong light, the diazo body will decompose and the eventual color will be spoiled. The diazotizing bath should smell distinctly of nitrous acid, and if such is not the case, more sodium nitrite and acid should be added. Care should be taken that this bath does not become heated by leaky steam-pipes, etc. Sometimes, in fact, ice is added to this bath for the purpose of keeping the temperature down (hence these colors are sometimes spoken of as "ice-colors"); but if the bath is kept at the ordinary temperature of water (about 60 to 70° F.) no artificial cooling is necessary. The third bath is termed the "developing" bath, and the beta-naphthol (or other like body) is spoken of as the "developer." Its function is to combine with the unstable diazo body to give the new and permanent coloring-matter. This bath should also be cold, otherwise the diazo body on first entering the bath will be decomposed before it has had a chance to become fixed by the developer. Beta-naphthol is not very soluble in water (especially cold water), hence, before adding it to the bath it is advisable to dissolve it in a little hot water together with its weight of soda ash, or caustic soda, and add this solution to the developing bath.

**Experiment 104. Showing the Action of Heat on the Diazo Body.** — Dye a skein of cotton yarn as before with 6 per cent. of Primuline; rinse and diazotize in a bath containing 5 per cent.



of sodium nitrite and 6 per cent. of sulphuric acid; work for 10 minutes at a temperature of 180° F., then rinse, and pass into the developing bath prepared as above described; work cold for 10 minutes, then wash and dry (351). Compare the color obtained on this skein with that on the one in the previous experiment. Dye another skein of cotton yarn with 6 per cent. of Primuline as before, and diazotize cold as in the previous experiment. Then wash the skein in hot water for 10 minutes, and afterwards develop as already described in the beta-naphthol bath cold for 10 minutes (352). Notice the influence of the hot washing on the eventual color. Dye another skein with 6 per cent. of Primuline as before; diazotize cold, and expose to the air for several hours; then develop as usual in the beta-naphthol bath cold for 10 minutes (353). Notice the influence of the long exposure on the color.

**Experiment 105. Developed Black on Cotton.** — Although there are several black dyes among the substantive colors, yet they do not yield very satisfactory colors either as regards depth of tone or fastness to bleeding when dyed directly. Some of these may be diazotized and developed, however, and so produce black colors of great beauty and fastness. Dye a skein of cotton yarn in a bath containing 6 per cent. of Diamine Black BH, 20 per cent. of common salt, and 1 per cent. of soda ash; enter at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour; rinse (354), and diazotize as usual, and then develop with 2 per cent. of phenylene-diamine in same manner as employed for beta-naphthol; wash well and dry (355). Phenylene-diamine is best dissolved previously to its addition to the bath in a little soda ash. Test the black thus obtained for fastness to washing and cross-dyeing. Also preserve a sample of the color before diazotization, and compare it in tone of color with the developed dyeing; also test the fastness of the direct dyeing to washing (356) and cross-dyeing (357), and compare these results with those for the developed dyeing.

**Experiment 106. Dyeing Primuline on Silk.** — Some of the developed dyes are very suitable for the dyeing of fast colors on



silk. Dye a skein of silk in a bath containing 10 per cent. of Glaubersalt and 10 per cent. of Primuline; enter at 140° F., and gradually bring to the boil and dye at that temperature for one-half hour; rinse, and diazotize and develop with beta-naphthol as already described in the foregoing experiments (358). This should give a good heavy red which is fast to washing and water.

**Experiment 107. Dyeing a Developed Black on Silk.** — Dye a skein of silk yarn in a bath containing 10 per cent. of Glaubersalt and 10 per cent. of Zambesi Black D in the usual manner; diazotize and develop with 3 per cent. of toluylene-diamine. Wash well and dry (359). Test this color for fastness to washing and water.

#### NOTES. .

**1. The Production of Developed Colors on Cotton.** — The defect of the substantive dyes on cotton is their liability to bleed when washed, although this may be remedied in some cases by an after-treatment with certain metallic salts; still faster dyeings may usually be obtained by the diazotizing and developing process. This process is more especially employed for the production of primuline red as a substitute for the more expensive Turkey-red, and for the production of fast blacks; the other colors are not so much used. The developing process not only materially increases the fastness of the colors to washing and acids but it also greatly increases the intensity of the shade. It has already been said, in fact, that the substantive colors do not yield very deep shades on cotton, even when large amounts of dyestuff are used in the bath; in many cases the blacks when dyed direct give only dark blue or slate colors, and only produce a deep black on being diazotized and developed. Not all of the substantive dyes may be developed, but a sufficient number of them are susceptible to this treatment to give a wide range of shades, and there are a number of the dyes which diazotizing does not affect, and which may in consequence be used for purposes of shading, being added directly to the same dye-bath as the developed color.

It must be borne in mind that developed colors require three different operations and as many different baths; this necessitates, of course, a triple handling of the cotton and therefore a greater expense than when dyeing the substantive colors alone. There is nothing different in the first dyeing process of the developed colors beyond that of the ordinary substantive dyes, which have already been discussed. The second operation, that of diazotizing, is the same for all the developed colors, and consists in working the dyed and rinsed cotton in a cold bath containing sodium nitrite and hydrochloric acid; the operation requires only 10 to 15 minutes. After diazotizing it is well to rinse the goods in water slightly acidulated with hydrochloric acid. The third operation, that of developing, is also done in a cold bath and requires only from 10 to 15 minutes; the kind of developer used depending on the dyestuff employed and the color desired. A diazotizing bath for 10 pounds of cotton can be prepared with  $\frac{1}{2}$  lb. sodium nitrite and  $\frac{3}{4}$  lb. hydrochloric acid (of 32° Tw.); sulphuric acid may be used in place of hydrochloric, in which case only  $\frac{1}{2}$  lb. of acid (of 168° Tw.) is employed. In preparing the bath the nitrite should first be dissolved in some water, added to the bath, after which the acid is added. For standing baths only one-third of the above-mentioned quantities is used. In order to ascertain if the diazotizing bath is still active, dip into it a piece of paper impregnated with starch paste and potassium iodide, which should at once turn blue. When working the bath it should smell distinctly of nitrous acid, though the odor should not be too pungent, which would indicate an excess of nitrite. This is not necessarily injurious, but should be avoided for reasons of economy. The diazotizing is best conducted in wooden vessels, though when dyeing in machines the diazotizing and developing may take place in copper vessels. It is not necessary to hydro-extract or wring out after diazotizing; the goods are allowed to drain, are then rinsed slightly in water acidulated with 1 pint of hydrochloric acid to 100 gallons, and then entered directly into the developing bath. It is also important to remember that the

diazotized goods should not be left standing for any length of time, but the rinsing and developing should proceed immediately after the diazotizing. Especial care should be taken not to expose the diazotized color to glaring light or to any source of heat. The developing bath is prepared with cold water and the requisite amount of developer in solution. The goods are turned a few times in this bath, then taken out and rinsed off. The beta-naphthol solution for developing may be prepared conveniently by dissolving 7 lbs. 3 ozs. of beta-naphthol and 6 lbs. caustic soda (of 77° Tw.) in 10 gallons of boiling water; for each 10 lbs. of cotton developed use 1½ pints of this solution. For phenylene-diamine or toluylene-diamine, dissolve 4½ lbs. of the salt with 1½ lbs. of soda ash in 10 gallons of boiling water, and 1½ pints of this solution is sufficient for 10 lbs. of cotton. If the baths are used repeatedly, the above quantities are used for the first two to three lots, after which only three-fourths the amounts are taken. The amount of water in the diazotizing and developing baths should be about 20 times the weight of the cotton. An addition of bluestone to the diazotizing bath increases the fastness to light of the color in most cases; for such purpose, however, it is best to give an after-treatment with bluestone after developing, by passing the goods through a cold or lukewarm bath containing 3 per cent. of bluestone and 3 per cent. of acetic acid, and then rinsing. After development or after-treatment the cotton is usually soaped or oiled for the purpose of softening. Developed dyeing may be topped with basic dyes in the same manner as the direct dyeings with substantive colors.

## 2. List of the Principal Developed Dyes.

Diamine Azo Black.	Diaminogene Blue.
Diamine Azo Blue.	Diaminogene extra.
Diamine Black.	Dianil Dark Blue.
Diamine Blue Black.	Diazo Black (all brands).
Diamine Bronze.	Diazo Blue.
Diamine Brown.	Diazo Blue Black.
Diamine Catechine.	Diazo Bordeaux.
Diaminogene.	Diazo Brown.



## LIST OF THE PRINCIPAL DEVELOPED DYES.—Continued.

Diazo Brilliant Black.	Oxamine Black A.
Diazo Dark Blue.	Oxamine Violet.
Diazo Fast Black.	Polychromine A and B.
Diazo Indigo Blue.	Primuline (all brands).
Diazo Rubine.	Thiochromogene.
Diazurine B.	Toluylene Blue Black.
Diazyl Black	Zambesi Black.
Direct Indigo Blue.	Zambesi Blue.
Hessian Bordeaux.	Zambesi Brown.
Indigo Blue B.	Zambesi Gray.
Melanogen Blue BH.	Zambesi Indigo Blue.
Melantherine.	

## SAMPLES.

348. Primuline before diazotizing.
349. Primuline after diazotizing.
350. Primuline after development.
351. Primuline when diazotized in hot bath.
352. Diazotized Primuline heated before development.
353. Diazotized Primuline exposed to light.
354. Diamine Black BH before dyed direct.
355. Diamine Black BH diazotized and developed.
356. Washing test for diazotized black, and before diazotizing.
357. Cross-dye test for diazotized black, and before diazotizing.
358. Primuline on silk.
359. Developed black on silk with Zambesi Black D.

## QUIZ 17.

505. What are developed or diazotized colors? To what general class of dyes do they belong?

506. What was the first developed dyestuff discovered? What is the general principle of dyeing the developed dyes?

507. What color does Primuline give when dyed as an ordinary substantive dye? What change in color is noticed on diazotizing? On developing?

508. Of what does the diazotizing bath consist? To what is the odor of this bath due? What chemical reaction takes place between the constituents of this bath?

509. What is the "amido" group? What is the action of nitrous acid on it? What is the "diazo" group?

510. Why is the diazotizing bath used cold? Why are the diazotized colors sometimes spoken of as "ice colors"?



511. What is the function of the developing bath? How is the developing bath with beta-naphthol prepared?

512. How long does the treatment last in the diazotizing and developing baths? Why should development immediately follow diazotizing?

513. What is the effect of heating the diazotizing bath? Of heating the diazotized color before development?

514. What is the effect of exposing the diazotized color to the action of light and air for some time previous to development?

515. Can a satisfactory black color, as a rule, be obtained with substantive dyes directly? Describe how a good black may be obtained with Diamine Black BH. What developer is employed?

516. What is the fastness of the developed Diamine Black to washing and cross-dyeing? How were these tests made?

517. Give the method for dyeing Primuline red on silk. Is this red fast to washing and water?

518. How did you produce a developed black on silk? What dyestuff and developer were used? What was the fastness of this color to washing and water?

519. What is the general object of diazotizing and developing the substantive colors? What are the principal colors obtained in this method?

520. May all substantive dyes be developed? How may the developed dyeings be shaded?

521. How many operations and baths do the developed colors require? Why are they more expensive than substantive colors dyed direct?

522. Does the diazotizing process differ with different colors? How should the dyed goods be rinsed after diazotizing? On what considerations does the selection of the proper developer depend?

523. How would you prepare a fresh diazotizing bath for 10 lbs. of cotton yarn? What amount of chemicals would you add to a standing kettle?

524. What acids may be used in the diazotizing bath, and what is the ratio of their proportions?

525. How would you test the diazotizing bath to ascertain if it still contained nitrous acid? On what chemical principle does this reaction depend?

526. In what kind of vats is it best to conduct the diazotizing process? May copper vessels be employed?

527. How would you prepare the acid wash water for rinsing off the diazotized color before development?

528. What precautions should be taken with the diazotized color previous to development in order to obtain good results?

529. How would you prepare the beta-naphthol solution of developer, and what quantity of this solution would be needed for 10 lbs. of cotton?

530. How are the solutions of phenylene- and toluylene-diamine prepared for a developer? What quantity is needed for 10 lbs. of cotton?

531. May the developing bath be used as a standing kettle? If so, what proportional amounts of developer are taken for succeeding baths?

532. What proportion of water should be employed in the diazotizing and developing baths?

533. How may an after-treatment with bluestone be given to developed dyeings? May developed dyeings be topped with basic dyes, and if so, in what manner?

## SECTION XVIII.

### SULPHUR DYES ON COTTON.

**Experiment 108. General Method of Applying Sulphur Dyes.** — Prepare a bath containing 5 per cent. of Immedial Brown B, 5 per cent. of sodium sulphide, 5 per cent. of soda ash, and 25 per cent. of common-salt. Dye a skein of cotton yarn in this bath, entering at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour; wash and dry (360). It will be noticed that the dye-bath does not exhaust very well, so dye a second skein (361) of cotton yarn in the same bath without adding any further dyestuff or chemicals, only diluting the bath to its original volume with water. Also dye a third skein (362) in the same manner. Compare the colors on the three skeins, the gradation of which will show the comparative exhaustion of the bath.

**Experiment 109. After-treatment of Sulphur Dyes with Chrome.** — An after-treatment with chrome is sometimes given in order to obtain a faster color. Dye a skein of cotton yarn with 5 per cent. of Immedial Brown B in the same manner as above described; squeeze, rinse, and treat in a fresh bath containing 2 per cent. of chrome and 3 per cent. of acetic acid; boil for 15 minutes; wash well and dry (363). Compare the color of this skein with the corresponding one in the previous experiment and note the effect of the after-treatment on the tone of the color. Also test the fastness of these two dyeings to washing and cross-dyeing (364, 365, 366, 367).

**Experiment 110. Obtaining Black with Sulphur Dyes.** — Dye a skein of cotton yarn in a bath containing 15 per cent. of Sulphur Black A extra, 15 per cent. of sodium sulphide, 5 per cent. of soda ash, and 50 per cent. of common-salt; enter at 140° F., gradually raise to the boil, and dye at that temperature for one-half hour; wash well, and treat in a bath containing 2 per cent. of olive oil

emulsion; then rinse and dry (368). Test the color of this skein for fastness to washing (369) and cross-dyeing (370).

**Experiment 111. Use of Autogene Black.** — This sulphur dye does not require the addition of sodium sulphide to the bath. Dye a skein of cotton yarn in a bath containing 50 per cent. of salt and 15 per cent. of Autogene Black; enter at 140° F., gradually raise to the boil, and dye at that temperature for three-quarters hour; wash well, and then soap in a warm dilute solution of soap; rinse and dry (371). This black gives a color much resembling Aniline Black. Test the fastness of the color to washing (372) and cross-dyeing (373). Compare the color of these sulphur blacks with that obtained with developed dyeings, and also compare the fastness of the colors.

#### NOTES.

1. **Use of the Sulphur Dyes.** — These colors belong to the general group of substantive cotton dyes and are of rather recent introduction. They are so called because they consist of sulphur compounds, and are dyed, as a rule, with the addition of sodium sulphide to the bath. The first of these dyes was discovered in 1867 by two French chemists, and was known as Cachou de Laval; it was prepared by fusing wood shavings and sawdust with sodium sulphide or sulphur. As it had but little tinctorial power it was not a success as a dyestuff. During the past decade, however, a large number of these dyes have appeared in almost all colors with the exception of red, and even during the past year a so-called sulphur red dye has been brought out, but it is far from being a pure red color. We have sulphur blacks, browns, blues, yellows, and greens. The sulphur colors are especially remarkable for their fastness to washing and even fulling, as well as acids in cross-dyeing. They also furnish deep, heavy shades on cotton. They are mostly dyed in a bath containing sodium sulphide, soda ash, common-salt, and many of them may be after-treated with chrome or bluestone with considerable improvement as to their fastness. The sodium sulphide is for the purpose of dissolving and in some cases of reducing the dyestuff (in the case



of certain blue dyes); the soda ash is for the purpose of correcting the hardness of the water and making the bath alkaline, as the dyes appear to work better in an alkaline bath; the salt is added as with ordinary substantive dyes, for the purpose of obtaining a better exhaustion of the dye-bath. In some cases the dyestuff itself contains sufficient sodium sulphide to dissolve it in the bath, and consequently none need be added. The sulphur dyes are evidently not as yet distinct chemical bodies; that is to say, the proper tinctorial principle has not been isolated from contaminating by-products in their manufacture, and consequently it takes a relatively large amount of dyestuff to obtain a full shade; from 10 to 15 per cent., as a rule, in the case of colors, and from 20 to 30 per cent. for blacks being required; in this respect, however, these dyes are constantly being improved, as better methods of manufacture are devised. At first, these dyes were sold usually in the form of irregular lumps which rapidly deteriorated on exposure to air and dampness with liberation of sulphuretted hydrogen; but in this respect there has been much improvement of late by selling the dyes in powder form mixed with proper drying substances to prevent the absorption of moisture on exposure. The sulphur dyes exhaust badly and require a large amount of salt in the bath where heavy shades are to be dyed.

The sulphur dyes are best dissolved in wooden vessels by pouring over them hot water containing a part of the sodium sulphide required for the dyeing. The dye-baths should be of wood, and all the metallic pipes and fittings should be of iron or lead, copper and brass being avoided as much as possible, as these have a bad effect on the dyes. The sodium sulphide in the bath should be sufficient to thoroughly dissolve all the dyestuff so that the bath is clear; if the bath is turbid, more sodium sulphide should be added. Unnecessary boiling of the dye-bath should be avoided, as this causes the oxidation of the sulphide to too great an extent; if there is too much sodium sulphide present, on the other hand, the cotton will not take up the color well and the dyeings will appear thin. In place of using common-salt, glauber-salt may be used with like effect. In order to control the amounts

of salts which are present in standing kettles it is best to use hydrometer tests; for blacks the cold dye liquor should stand at 8 to 10° Tw., but for blues and other colors it should not exceed 4 to 5° Tw. It should be borne in mind that 10 parts of common-salt are equivalent to 12 parts of calcined glaubersalt or to 24 parts of the crystallized glaubersalt. In special cases glucose, dextrin and Turkey-oil red are added to the bath in order to secure better exhaustion and better penetration of the goods. In nearly all cases the sulphur dyes may be dyed in a boiling bath, though just under the boil is a better practice; in the case of some blue dyes, the temperature of the bath should not be over 85° F. The sulphur colors may also be dyed very well in lukewarm or even cold baths. After dyeing it is important that the goods be well squeezed and thoroughly rinsed immediately after coming from the dye-bath, in order to prevent the precipitation of unfixed dye-stuff superficially on the fibre; this gives rise to more even shades and the colors are faster to rubbing. With some of the blues it is necessary to oxidize the color after dyeing; this may be done by squeezing and hanging in the air, or by steaming in the air.

## 2. List of the Principal Sulphur Dyes.

### (a) ORANGE.

Eclipse Orange.	Pyrogene Orange
Immedial Orange.	Thiogene Orange.

### (b) YELLOW.

Eclipse Yellow.	Pyrogene Yellow.
Immedial Yellow.	Thiogene Golden Yellow.
Kryogene Yellow.	Thiogene Yellow.

### (c) GREEN.

Eclipse Green.	Katigene Olive.
Eclipse Olive.	Kryogene Olive.
Immedial Green.	Nigrosulphine.
Immedial Olive.	Pyrogene Olive.
Katigene Chrome Blue 5G.	Pyrogene Green.
Katigene Green.	Thiogene Green.

*(d)* BLUE.

Eclipse Blue.	Pyrogene Blue.
Immedial Blue.	Pyrogene Direct Blue.
Immedial Direct Blue.	Pyrogene Indigo Blue.
Immedial Indone.	Sulphur Blue.
Immedial Pure Blue.	Thiogene Blue.
Immedial Pure Blue.	Thiogene Cyanine.
Immedial Sky Blue.	Thiogene Dark Blue.
Katigene Chrome Blue.	Thiogene Direct Blue.
Katigene Indigo.	Thiogene Violet.
Kryogene Blue.	Thion Blue.
Melanogene Blue.	

*(e)* BROWN.

Cachou de Laval.	Pyrol Brown.
Eclipse Bronze.	Sulphanil Brown.
Eclipse Brown.	Sulphogene Brown.
Immedial Bronze.	Sulphur Brown.
Immedial Brown.	Thiocatechine.
Immedial Dark Brown.	Thiogene Brown.
Katigene Black Brown.	Thiogene Catechu.
Katigene Brown.	Thiogene Dark Red.
Katigene Chrome Brown.	Thiogene Khaki.
Katigene Yellow Brown.	Thion Brown.
Kryogene Brown.	Vulcan Brown.
Pyrogene Brown.	

*(f)* BLACK.

Anthraquinone Black.	Pyrol Black.
Auronal Black.	Sulphanil Black.
Autogene Black.	Sulphur Black.
Cross-Dye Black	Sulphur Blue Black.
Eclipse Black.	Thiocarbene.
Immedial Black.	Thiogene Black.
Katigene Black.	Thiogene Black Liquid.
Katigene Blue Black.	Thiogene Coal Black.
Kryogene Black.	Thiogene Diamond Black.
Melanogene.	Thion Black.
Mercaptol Black.	Thional Black.
Pyrogene Black.	Thiophenol Black.
Pyrogene Gray.	Vidal Black.

**SAMPLES.**

- 360. Immedial Brown on cotton; general method; first bath.
- 361. Immedial Brown; second bath.
- 362. Immedial Brown; third bath.
- 363. Immedial Brown, after-treated with chrome.
- 364. Washing test Immedial Brown before chroming.
- 365. Washing test Immedial Brown after chroming.
- 366. Cross-dye test Immedial Brown before chroming.
- 367. Cross-dye test Immedial Brown after chroming.
- 368. Sulphur Black A extra on cotton.
- 369. Washing test Sulphur Black A extra.
- 370. Cross-dye test Sulphur Black A extra.
- 371. Autogene Black on cotton.
- 372. Washing test Autogene Black.
- 373. Cross-dye test Autogene Black.

**QUIZ 18.**

- 534. Give the general method for the dyeing of sulphur colors on cotton.
- 535. Does the dye-bath with sulphur colors exhaust well? How may this be shown?
- 536. What is the purpose of after-treating sulphur dyes with chrome? How is the process carried out?
- 537. How was the color of Immedial Brown affected by the treatment with chrome?
- 538. What difference was noticed in the fastness of the untreated and the chromed dyeings with Immedial Brown as to washing and cross-dyeing? How were these tests carried out?
- 539. How is Sulphur Black A extra dyed on cotton? What character of black does it yield? What is the purpose of oiling the cotton after dyeing?
- 540. What is the fastness of Sulphur Black A extra to washing and cross-dyeing?
- 541. How does the dyeing of Autogene Black differ from that of the previous color? What is the character of the black obtained? What is its fastness to washing and cross-dyeing?
- 542. How do the color and the fastness of the sulphur blacks compare with those of the developed blacks?
- 543. To what general group do the sulphur colors belong? What was the first sulphur dye discovered? How was it prepared?
- 544. Have the sulphur colors been in general use for any length of time? Why are they called sulphur colors?
- 545. Through what range of colors do the sulphur dyes at present extend?
- 546. By what properties are the sulphur dyes characterized?



547. What substances are usually added to the dye-bath in using sulphur dyes? Give the function of each.

548. With what substances may the sulphur dyes be after-treated?

549. Do all sulphur dyes require the addition of sodium sulphide to the bath?

550. About what amounts of dyestuff are required for the production of full shades in dyeing with sulphur colors?

551. How should the sulphur dyes be stored? How are they affected by moist air?

552. Why is such a large amount of salt used in dyeing the sulphur colors?

553. What is the best manner of dissolving the sulphur dyes? Of what should the dye-vats consist, and what metals should be absent? What metals may be used for fittings?

554. How should the amount of sodium sulphide in the bath of sulphur dyes be regulated? What does turbidity in the bath indicate?

555. Why should excessive boiling of the dye-bath be avoided when using sulphur colors?

556. If too much sodium sulphide is present in the sulphur color bath what defect will be the result?

557. In the sulphur color dye-bath the common-salt may be replaced by what other salt?

558. How should the amounts of salts present in standing kettles of the sulphur colors be regulated? What should be the density of the liquors for blacks? For colors?

559. How many parts of calcined glaубersalt are equivalent to 100 parts of common-salt? What is the proportion between calcined glaубersalt and crystallized glaубersalt?

560. What is the difference as to composition between the calcined and the crystallized glaубersalts?

561. What other additions are sometimes made to the sulphur color dye-bath besides those already mentioned in order to obtain better exhaustion and penetration?

562. At what temperature is it best to dye the sulphur colors? Are boiling baths necessary?

563. Why is it important to thoroughly wash the goods after coming from the sulphur dye-bath? What after-treatment is necessary for certain blue sulphur dyes, and how is this after-treatment conducted?

## SECTION XIX.

### USE OF LOGWOOD IN DYEING.

**Experiment 112. General Method of Dyeing Logwood on Wool.** — Mordant four test-skeins of woolen yarn in the usual manner with 3 per cent. of chrome and 4 per cent. of tartar; wash well. Dye the first skein in a bath containing 2 per cent. of logwood extract (solid); enter at 140° F., gradually raise to the boil, and dye at that temperature for three-quarters hour, then wash well and dry (374). Dye the second skein in a bath containing 5 per cent. of logwood extract in the same manner (375). Dye the third skein in the same way with 15 per cent. of logwood extract (376). The lower percentages of logwood give bluish shades on a chrome mordant, which deepen into a bluish black in the heavy percentage. Dye the fourth skein in a bath containing a decoction made by boiling 50 per cent. of chipped logwood in 300 cc. of water (377). Notice that at first the dye-bath is of a reddish color, but that the black develops on boiling.

**Experiment 113. Effect of Over-Chroming.** — Mordant a test-skein of woolen yarn with 10 per cent. of chrome and 4 per cent. of tartar in the usual manner; wash well, and dye in the manner described above with 15 per cent. of logwood extract (378). It will be found that only a gray color is produced; this is the result of employing too much chrome, whereby the fibre becomes oxidized and loses its affinity for the dyestuff. It may also be probable that the excess of chrome has some injurious action on the logwood itself.

**Experiment 114. Shading Logwood with Alizarin Yellow.** — This is for the purpose of obtaining a deep black without the bluish tone of the straight logwood black. Mordant a skein of woolen yarn in the usual manner with 3 per cent. of chrome and 4 per cent. of tartar; wash, and dye in a bath containing 15 per

cent. of logwood extract and  $\frac{1}{4}$  per cent. of Alizarin Yellow AW. Enter at 100° F., gradually raise to the boil, and dye at that temperature for three-quarters hour; then wash well and dry (379). Compare the color of this skein with that dyed with logwood alone.

**Experiment 115. Logwood Black on Cotton with an Iron Mordant.** — The principal use of logwood on cotton is for the production of blacks and grays in connection with an iron mordant. The chief salt used for this purpose is the so-called "nitrate of iron." The mordant is fixed by means of tannin. Steep a test-skein of cotton yarn in a bath containing 4 per cent. of tannic acid; enter at 180° F., work for 15 minutes at that temperature, then allow to steep under the liquor without further heating for 1 hour. Squeeze, and pass through a bath of nitrate of iron at 4° Tw. for 15 minutes cold; then squeeze and pass through a weak bath of lime-water cold for 10 minutes, and finally wash well (380). The tannate of iron thus formed on the fibre imparts to the cotton a dark gray color. Now dye the skein in a bath containing 15 per cent. of logwood extract (solid) and 2 per cent. soda ash; entering at 160° F., gradually raise to the boil, and dye at that temperature for three-quarters of an hour. Wash well and dry (381).

**Experiment 116. To Obtain a Faster and Clearer Black.** — Mordant a test-skein of cotton yarn in the same manner as above with 4 per cent. of tannic acid, and then fix by passing through the baths of nitrate of iron and lime-water. Then dye as before with 15 per cent. of logwood extract. After dyeing, work the skein in a bath containing 1 per cent. of chrome; enter at 180° F., work for 15 minutes at that temperature, then squeeze and wash well, and finally soap off in a warm dilute soap bath (382). Instead of using the chrome bath the dyed material may be passed back into the nitrate of iron bath. This after-treatment and scouring with soap have the effect of preventing the rusty appearance liable to develop when logwood is dyed with an iron mordant. It is sometimes the practice to pass the cotton through a weak lime bath after coming from the tannin bath and before



entering the bath of nitrate of iron; this causes the formation of tannate of lime, and prevents a large amount of the unfixed tannin from passing into the iron bath and precipitating tannate of iron.

**Experiment 117. Dyeing Logwood without Tannin.** — Cotton may also be dyed with iron and logwood without the intervention of tannin. Steep a test-skein of cotton yarn in a bath containing nitrate of iron at 8° Tw., cold, for one-half hour; squeeze and work for 15 minutes in a bath containing 5 grams of soda ash at 140° F. This causes a precipitation of ferric oxide or iron buff in the fibre (383). Wash, and dye as previously described in experiment 1 with 15 per cent. of logwood extract (384).

**Experiment 118. Dyeing Silk a Pure Black with Logwood.** — By "pure" black is meant one which does not contain any weighting materials. Mordant a test-skein of silk yarn in a bath containing 150 cc. of water and 20 per cent. of cutch; enter at 120° F., and gradually bring to the boil, then allow to cool in the bath for one-half hour; next rinse the skein slightly and pass into a bath of nitrate of iron at 10° Tw., work at 120° F. for 15 minutes, then pass through a dilute bath of soda ash, and wash well (385). Next dye in a bath containing 150 cc. of water and 25 per cent. of logwood extract (solid) and 5 per cent. of soda ash; enter at 140° F., gradually bring to the boil, and dye at that temperature for one-half hour, then wash well and dry (386).

#### NOTES.

1. **Logwood.** — Logwood is obtained from the Campeachy wood, known botanically as *Hæmatoxylon Campechianum*; it is a large tree and grows principally in tropical and sub-tropical America. The wood itself is really a red-wood, but the color-lake as finally developed is black or blue, depending on its intensity. When freshly cut the wood is colorless or looks about like that of any other tree; on exposure to the influence of the oxygen of the air, however, the outside of the wood becomes of a dark reddish brown color, due to the development of the coloring-matter. The coloring principle of logwood is called hæmatoxylin,



and this on oxidation yields hematin, which is the real coloring-matter of the prepared logwood. In order to prepare the wood for use by the dyer, the logs, after having the outer sapwood stripped off, are either rasped or chipped, the chips being placed in large heaps and moistened with water. These heaps are turned over from time to time to allow the oxygen of the air free access to the wood. Fermentation occurs, which results in the formation of the hematin. The dyewood in this state may now be used by the dyer, but at the present time it is customary to carry the manufacture of the dyestuff still further and prepare an extract either in the solid or the liquid form.

Logwood is about the only natural coloring-matter which is still extensively used (with the exception of indigo). Its principal use at the present time is for the black dyeing of silk and leather; its use on cotton is decreasing, and on wool it is used only for very cheap blacks. This is due to the fact that there are several blacks for both wool and cotton which are much faster than logwood. On wool logwood is almost entirely used on a chrome mordant, and the color obtained is a bluish-black. About 15 per cent. of logwood extract is required for the production of full shades. To neutralize the bluish tone of the straight logwood, it was formerly the custom to use some fustic (a yellow wood color) in connection with the logwood. Fustic is still used in this manner, but as it is rather fugitive, it is better to employ a faster mordant yellow dyestuff for this purpose. In the dyeing of logwood it is to be noticed that an excess of chrome in the mordanting bath is injurious to the color. Sometimes logwood black on wool is after-chromed for the purpose of making the color faster to washing and fulling. Stannous chloride is at times added to the dye-bath for the purpose of giving a violet tone to the black. Logwood extract is sometimes mixed with copperas and blue-stone and sold in the form of a paste as a direct logwood black for wool; it is dissolved by adding oxalic acid to the bath. A direct chrome black can also be prepared by precipitating a solution of logwood with chrome and dissolving the precipitate in oxalic acid. Sometimes wool is first dyed in the indigo vat to

a blue, and then topped off with logwood, giving a bluish black; this is known as a "woaded" black.

Logwood is chiefly dyed on cotton in connection with an iron mordant, "nitrate of iron" being principally used. The iron salt is generally fixed on the cotton by tannin preliminary to dyeing, but at times the fixation of the iron is accomplished by the tannin naturally present in the logwood extract. As the iron mordant gives a brownish or rusty black, it is advisable to chrome the color obtained to produce a clearer and more desirable black. The development of the rusty appearance on an iron-logwood black on cotton may also be prevented more or less by soaping the dyed color. By the addition of bluestone to the dye-bath the color is also said to be improved in appearance.

Logwood is still used extensively for the black dyeing of silk, both unweighted and weighted. It is used in connection with a tannin-iron mordant, the tannin employed usually being cutch. Logwood seems to make the silk fibre opaque, which is a necessary condition for the production of a full deep black; the coal-tar blacks, as a rule, do not make the silk sufficiently opaque.

**2. General Use of Natural Dyes.** — Previous to the discovery of the coal-tar dyes the textile colorist had to rely upon either the mineral pigments or the dyestuffs derived from the various vegetable substances for the production of his effects. The vegetable dyes nearly all belong to the mordant class of dyestuffs, though a few such as turmeric, safflower, and annatto exhibit substantive properties to a certain degree, and may be dyed directly on cotton. In general, however, in using the natural dyewoods on either wool or cotton it is first necessary to mordant the material in the usual manner with metallic salts.

The coloring-matters present in the dyewoods were usually extracted by the dyer himself by simply boiling the rasped wood in water and using this solution as a dye-bath. Under these conditions, however, the coloring-matter so obtained was never in a pure condition, but was contaminated with more or less resinous and tannin matter which acted frequently in a deleterious manner in the dyeing. The first application of chemical

science to the art of dyeing was the attempt to manufacture purer and more homogeneous dye products from the extracts of the various dyewoods or other vegetable coloring-matters. At the present time the use of the natural dyewoods has almost disappeared with the exception of logwood, fustic, and indigo; and even the latter is now a coal-tar product which is rapidly driving the natural article from the market. Logwood still holds its own for the production of cheap blacks on wool and cotton, and it is also largely used in the black dyeing of silk. Fustic is used to some extent in connection with the foregoing to tone the shade of the black obtained, but even its use in this manner is growing less and less, being replaced by other yellow coloring-matters which possess a greater degree of fastness. Cutch is still used for the production of brown shades on cotton, but it is more used as a tannin mordant than as a self color.

The natural dyewoods yield coloring-matters from which may be dyed black, red, brown, yellow, blue, violet, etc.; there is, however, no good green dye among the list of natural dyestuffs. The colors obtained with the natural dyes, as a rule, are rather dull in appearance, and many of them are of questionable fastness, there being many of the mordant coal-tar dyes which are far superior in this respect.

#### SAMPLES.

- 374. Wool dyed with 2 per cent. logwood on chrome mordant.
- 375. Dyeing with 5 per cent. logwood.
- 376. Dyeing with 15 per cent. logwood.
- 377. Dyeing with 50 per cent. logwood chips.
- 378. Showing effect of over-chroming.
- 379. Logwood shaded with Alizarin Yellow.
- 380. Cotton mordanted with tannin-iron for logwood.
- 381. Cotton dyed with logwood on iron mordant.
- 382. Logwood on cotton after-chromed.
- 383. Cotton mordanted with iron oxide for logwood.
- 384. Cotton dyed with logwood without tannin mordant.
- 385. Silk mordanted with cutch and iron for logwood.
- 386. Silk dyed with logwood on tannin-iron mordant.



## QUIZ 19.

564. What is the general method for the dyeing of logwood on wool?
565. What mordant is principally used in the dyeing of logwood on wool?
566. What color do small percentages of logwood give on a chrome mordant? What color do heavy percentages give?
567. How much solid logwood extract would be required to dye a black on 100 lbs. of wool?
568. In dyeing with logwood chips how is the bath prepared? What amount of logwood is required to give a black when used in this form?
569. What is the color of the solution of logwood? Describe the changes in color of the wool as the dyeing proceeds.
570. What is the effect on the logwood black when too much chrome is used as the mordant? To what causes may this be due? What is the proper amount of chrome to use?
571. What tone of black does a straight logwood give on a chrome mordant? How may such a black be shaded in order to produce a dead black?
572. Explain why the addition of a small amount of yellow dyestuff to the logwood bath produces a fuller shade of black. What dyes may be used for this purpose?
573. What is the general method for the dyeing of logwood black on cotton?
574. What is the chief salt of iron employed for mordanting cotton for logwood black?
575. Of what does the so-called "nitrate of iron" consist? Why is it given this name?
576. How is the iron mordant fixed on the cotton when using "nitrate of iron"? What is the color of the mordanted cotton?
577. What is the purpose of treating the mordanted cotton with a bath of lime-water? What salt of iron is formed in the fibre when mordanting with iron and tannin?
578. How is the dye-bath prepared for dyeing cotton black with logwood on an iron mordant? How much logwood extract is required to give a black?
579. How may the rusty appearance of the logwood-iron black on cotton be prevented? Give the reasons for the processes employed.
580. What is the purpose of passing the tanned cotton through lime-water before mordanting with iron liquor? Explain the reaction which takes place.
581. How may an iron-logwood black be dyed on cotton without the use of a preliminary tannin bath? How is the iron mordant fixed, and what is the color of the mordanted cotton?
582. Give the general method for the dyeing of silk with logwood.
583. What is meant by a "pure" black on silk? By a "weighted" black?
584. What is the mordant employed in dyeing silk with logwood? What tannin is generally used for fixing?



585. Of what does the dye-bath consist when dyeing logwood black on silk? What is the color of the mordanted silk before dyeing?

586. Why is logwood so called, and from what is it obtained? What is its botanical name? Where does logwood grow?

587. To what class of dyewoods does logwood really belong? What is the nature of the color-lake formed with metallic mordants?

588. What is the color of logwood when freshly cut? What change occurs when exposed to the air?

589. What is the coloring principle of logwood? What does this yield on oxidation?

590. How is logwood prepared for use in dyeing? Are logwood chips much used at present by the dyer?

591. What position does logwood hold among the natural dyestuffs? What is its principal use at the present time?

592. Why has the use of logwood for the dyeing of wool and cotton diminished of late years?

593. What is the use of fustic in connection with logwood dyeing? Why is fustic not used so much at present?

594. What is the purpose of after-chroming logwood black on wool? What is the effect of using tin salts in the dye-bath with logwood?

595. Of what does the "direct logwood black for wool" consist? How is it dyed?

596. How may a direct chrome logwood black for wool be prepared? What is meant by a "woaded" black on wool?

597. What is the defect of the iron-logwood black dyed on cotton? How may this be overcome?

598. What is the effect of adding bluestone to the dye-bath of logwood on cotton?

599. In what manner does logwood black on silk differ from the general run of coal-tar blacks?

## SECTION XX.

### THE MINOR NATURAL DYES.

**Experiment 119. Use of Fustic.** — Mordant a test-skein of woolen yarn with 3 per cent. of chrome and 4 per cent. of tartar in the usual manner. Dye for 45 minutes in a bath containing 10 per cent. of fustic extract, entering at 140° F. and gradually raising to the boil. Wash well and dry (387). Mordant a second skein with 5 per cent. of stannous chloride and 5 per cent. of oxalic acid, and dye in the same manner as above with 10 per cent. of fustic extract (388). Mordant a third skein with 6 per cent. of ferrous sulphate and 8 per cent. of tartar, and dye as before with 10 per cent. of fustic extract (389). Note the difference in color obtained from the fustic by the use of different mordants. Test the fastness of the color to washing and light, and compare the results with those given by Alizarin Yellow.

**Experiment 120. Use of Madder.** — This dyestuff was formerly very extensively employed, but has now been replaced almost entirely by the synthetically prepared Alizarin which is the coloring principle of madder. Madder consists of the ground root of *Rubia tinctorum*, and is applied as a mordant dyestuff. Mordant a test-skein of woolen yarn with 3 per cent. of chrome and 4 per cent. of tartar, and dye for 45 minutes in a bath prepared by boiling 25 per cent. of madder in water and straining. Also add 4 per cent. of calcium acetate to the dye-bath. This mordant yields a reddish brown color with madder (390). In a similar manner dye a test-skein which has been mordanted with 10 per cent. of aluminium sulphate and 8 per cent. of tartar (391). This mordant yields a dull red color. Dye a third test-skein mordanted with 5 per cent. of stannous chloride and 5 per cent. of oxalic acid, and notice that an orange-red color is obtained (392).

**Experiment 121. Use of Archil.** — This dye, together with the related coloring-matter cudbear, is but little used at present. It possesses the character of a substantive dye towards wool and yields a dull magenta shade. It can be applied in a neutral bath, though the addition of acid makes the color redder and brighter. The color is not fast to light or fulling, and only fairly so to washing. Dye a test-skein of woolen yarn in a neutral bath containing 20 per cent. of archil paste, entering at 120° F. and raising to the boil for 45 minutes (393). Dye a second skein in a bath containing 20 per cent. of archil paste and 4 per cent. of sulphuric acid (394). Notice the difference in the color caused by the use of the acid in the dye-bath. Archil at the present time is not used as a self color, but in combination with various acid dyes for the production of browns, maroons, and clarets; it is also employed as a bottom for indigo. It is not used for the dyeing of cotton. Silk may be dyed with archil in a soap bath, with or without the addition of acetic acid.

**Experiment 122. Use of Quercitron.** — This is a yellow coloring-matter obtained from the bark of a species of oak. Quercitron itself consists of the ground bark, while *flavine* is the pure dry extract of the coloring-matter. Mordant a test-skein of woolen yarn with 3 per cent. of chrome and 4 per cent. of tartar in the usual manner, and then dye with 10 per cent. of quercitron bark, boiling for 45 minutes (395). On this mordant quercitron gives an olive-yellow color. Mordant another test-skein with 4 per cent. of stannous chloride and 2 per cent. of oxalic acid; remove the skein from the mordanting bath, add 1 per cent. of flavine extract, boil up for 5 minutes, then reënter the wool and continue boiling for 45 minutes (396). This method of dyeing should yield a bright canary-yellow. By increasing the amount of flavine the color becomes orange-yellow. Quercitron and its products are but little used for wool dyeing at present, though they are still employed to some extent in both cotton and wool printing. The color is not particularly fast to either light or scouring.

**Experiment 123. Use of Cutch.** — This brown dyestuff is also a tannin, and in this latter connection it has already been con-



sidered (see page 127). As a dyestuff it was formerly very extensively used on cotton, and for the production of certain tones of brown it is still employed quite largely in cotton dyeing. It is not used for the dyeing of wool, as the fibre is made too harsh. In silk dyeing it is largely used, but only as a tannin in connection with logwood black. Prepare a bath containing 15 per cent. of cutch and dye a test-skein of cotton yarn for 1 hour at 195° to 210° F. Then squeeze the skein and treat for 30 minutes at 160° F. in a bath containing 5 per cent. of chrome, and finally give a thorough washing (397). Darker shades of brown are obtained by the addition of bluestone to the dye-bath as follows: Dye a test-skein of cotton yarn in a bath containing 15 per cent. of cutch and 2 per cent. of bluestone; squeeze and treat with chrome solution as above (398).

**Experiment 124. Use of Cochineal.** — This dyestuff consists of the dried bodies of small insects and furnishes a bright red coloring-matter. Before the introduction of the red acid dyes it was largely used for the production of bright scarlet colors on wool, and it still has a certain degree of use for this purpose. Mordant a test-skein of woollen yarn with 6 per cent. of stannous chloride and 4 per cent. of oxalic acid; wash, and dye in a fresh bath containing 10 per cent. of ground cochineal, starting at a temperature of 140° F. and raising to the boil for 45 minutes (399). When dyed on a tin mordant in this manner a bright scarlet color is obtained which is fast to light and fulling. A crimson-red color is obtained with an aluminium mordant as follows: Mordant a test-skein of woollen yarn with 8 per cent. of aluminium sulphate and 6 per cent. of tartar; wash, and dye in a fresh bath containing 10 per cent. of ground cochineal (400). Cochineal is not used for the dyeing of cotton.

#### NOTES.

1. **Fustic.** — This coloring-matter is obtained from the wood of a tree botanically known as *Morus tinctoria*. It is also known as Cuba wood or yellow wood. It is obtained in the West Indies and Central and South America, the best varieties being obtained



from Cuba and Tampico. Fustic gives a bright yellow color with an alum mordant, with chromium an olive-yellow, with iron a dark olive, with copper an olive, and with tin a bright orange-yellow. Fustic may be used either in the form of the chipped wood or as the extract, the latter being obtainable either as paste or solid. At present it is seldom used as a self color, but it still finds considerable use in connection with logwood for the dyeing of dead-black shades.

Fustic appears to contain two coloring-matters, morintannic acid and morin. The former, known also as maclurin, is readily soluble in water, and may be crystallized from solution in the form of light yellow microscopic needles. It has the composition  $C_{13}H_{10}O_6$ ; when heated with strong caustic alkali it is decomposed into phloroglucin and protocatechuic acid. It dissolves in cold concentrated sulphuric acid with a yellow color and is reprecipitated on dilution with water. If the strong acid solution is allowed to stand for some days it deposits brick-red crystals of rufimoric acid. If a solution of morintannic acid is treated with zinc and sulphuric acid, the solution becomes red and then orange, and contains phloroglucin and machromin; the latter crystallizes in slender needles which become blue on exposure to the air. Hydrochloric acid gives a blue precipitate, and the alkaline solution also becomes blue on exposure to the air. A solution of machromin with ferric chloride gives a violet color gradually becoming blue; mercuric chloride gives the same result. A solution of morintannic acid with gelatin gives a yellow precipitate, with ferro-ferric sulphate a greenish precipitate, acetate of lead a yellow precipitate, and stannous chloride an orange precipitate. Morin, on the other hand, is almost insoluble in cold water, and only slightly soluble in boiling water. It is also known as moric acid, and has the formula  $C_{12}H_{10}O_6$ . It is soluble in alkalies, with a yellow color, from which solution it is reprecipitated by the addition of acids. It is soluble in alcohol, and this solution with ferric chloride gives an olive-green color. Morin appears to give much deeper shades with chromium and aluminium mordants than morintannic acid, but it gives lighter shades

with iron mordants. Morintannic acid may be prepared from commercial fustic extract by allowing the concentrated syrupy solution to stand for some days, when an abundant crystalline deposit will be formed; this is washed rapidly with a little cold water and strongly pressed. The resulting mass is boiled twice with water, whereon a solution containing morintannic acid will be obtained, the residue consisting of moric acid and morate of lime. The aqueous solution is concentrated by evaporation, and precipitated by the addition of hydrochloric acid. Pure moric acid may be obtained from the residue by treating with dilute hydrochloric acid (to decompose the calcium morate) and dissolving in alcohol. On diluting with water this solution deposits moric acid in the form of yellow needles.

A solution of fustic gives the following reactions:

*Alkalies:* Orange to brown color.

*Weak acids:* Pale yellow precipitate.

*Alum:* Bright yellow precipitate.

*Lead acetate:* Orange precipitate.

*Copper acetate:* Brownish yellow precipitate.

*Ferrous sulphate:* } At first olive color, then brownish olive  
*Ferric sulphate:* } precipitate on standing.

*Stannous chloride:* Brownish yellow precipitate.

*Copper sulphate:* Dark green precipitate.

*Gelatin:* Yellow flocculent precipitate.

Fustic occurs in commerce in the form of log, chipped, rasped, ground, or as an extract. The extract is frequently sophisticated with glucose and quercitron bark extract, and varies in its specific gravity from 40° to 51° Tw. The specific gravity, however, like that of logwood, is of no guide to its value, on account of its being increased by the addition of adulterants. The best method of making a valuation of fustic in any of its forms is to conduct a series of comparative dye-tests on skeins of woolen yarn which have been previously mordanted with 3 per cent. of chrome and 4 per cent. of tartar, as in the case of logwood, or with 3 per cent. of stannous chloride and 5 per

cent. of oxalic acid. The tests are carried out in the usual manner.

Fustic is more used on wool than on cotton, and the general mordant employed is chrome, though when bright yellow colors are desired aluminium or tin mordants are used. The color obtained with the latter, however, is not very fast to washing and is quite fugitive to light, becoming duller and browner on exposure. Even on a chrome mordant, however, the color cannot be classified as fast to light. In the dyeing of fustic prolonged boiling must be avoided, as this causes the color to be dull and brownish, probably due to the presence of considerable tannin matter in the dyestuff which by protracted boiling and oxidation suffers decomposition into brownish coloring-matters. The addition of some glue solution to the dye-bath is said to obviate this defect.

Another coloring-matter similar to fustic, and which once had considerable use, is the so-called *young fustic*. This dyestuff consists of the ground wood of the sumac tree, *Rhus cotinus*. It was employed in practically the same manner as fustic (which was known as *old fustic*), but was a much inferior dyestuff, owing to its fugitive character. It has now practically disappeared from trade.

**2. Madder.** — This dyestuff was formerly of very great importance, and was largely cultivated in the southern part of Europe and Asia Minor. It was used for the production of Turkey-red on cotton and the dyeing of red on wool. Madder, however, has long been replaced by the coal-tar *alizarin*, which is identical in composition and properties to the natural product.

Madder is the ground root of the plant known as *Rubia tinctorum*; the principal coloring-matters yielded by the madder root are *alizarin*, *purpurin*, *pseudo-purpurin*, *xanthin*, and *chlorogenin*; the aqueous extract also contains from 10 to 15 per cent. of *sugar*. The most important of these constituents is the *alizarin*; the other coloring-matters, especially the *xanthin* and *chlorogenin*, have a deleterious effect in dulling the color produced by the *alizarin*. The coloring-matters exist in the root in the form of



glucosides, which are split up into the dyestuffs and a sugar through the action of a particular ferment.

Alizarin, which is the principal coloring-matter of the madder, may be obtained therefrom by extracting the ground root with alcohol, evaporating the solution to dryness, powdering the residue, spreading it on a filter paper on a heated plate; the extract melts and the paper absorbs the brown resinous matters, while the alizarin sublimes on the surface of the mass as large orange-red crystals.

Alizarin possesses the characteristics of a phenol, and is readily soluble in alkalies, with red color; it is only slightly soluble in boiling water, with a yellow color. Its solution gives the following reactions:

*Alkalies:* Bluish claret color.

*Acids:* Brownish yellow color.

*Alum:* Brownish red precipitate.

*Stannous chloride:* Brownish red precipitate.

*Iron salts:* Dark brown precipitate.

*Copper salts:* Reddish brown precipitate.

*Barium and calcium chlorides:* Violet precipitates.

*Lead acetate:* Reddish violet precipitate.

When alizarin is distilled with zinc it gives anthracene, from which reaction its synthetic preparation from the latter body was finally discovered.

*Purpurin*, which also exists in the madder root, resembles alizarin, but is more yellow in color. It may be prepared from alizarin by heating the latter with manganese dioxide and sulphuric acid.

Madder was used principally in the form of the ground root, but there were also numerous extracts and preparations made for the use of the dyer. *Garancin* was obtained by treating the wet paste of madder with concentrated sulphuric acid; 100 parts of madder gave from 30 to 40 parts of garancin, but this possessed four to five times the dyeing power of the original madder. It is supposed that other bodies which detracted from the good color



of the alizarin in the madder were removed by this treatment, and also any coloring-matter which may have been combined as metallic salts was liberated and rendered more active in dyeing. *Garanceux* was obtained from spent madder by the same process as the above; its coloring power, however, was only about one-third that of good garancin. *Fleurs de garance*, or *flowers of madder*, was prepared by treating madder with dilute sulphuric acid, whereby any yellow coloring-matters were removed. To prepare it, mix 100 parts of madder with 1 part sulphuric acid and 1000 parts water, and allow the mixture to macerate for 10 hours; filter, wash the paste, press and dry. The acid liquors from this process were used for the manufacture of alcohol, as they contained considerable sugar; 100 parts madder yielded about 10 parts alcohol.

Madder gives the following colors with the different mordants:

*Chromium*: Bluish red to crimson.

*Aluminium*: Pink to scarlet.

*Iron*: Maroon to reddish brown.

*Copper*: Yellowish brown.

*Tin*: Reddish orange.

Madder is still used in the woad indigo vat, but in this case it is more employed for its fermenting properties than for any coloring power.

The chief varieties of madder are Dutch, Alsatian, Avignon, and Turkish. Dutch madder is coarsely ground, and if kept in a moist place tends to cake together. *Crop madder*, which is the ground inner portion of the root, is considered as the best quality, while the outer part is known as *mulle madder*, and is the poorest. Alsatian madder is very like the Dutch. Avignon madder is known in two varieties, the *palus* and the *rosée*. The former is much the darker in appearance, due to the nature of the soil on which it is grown. Avignon madder does not require to be matured by storage for as long a time as Dutch and Alsatian madders, which should be stored in casks for two years before use. Turkish madder is exported chiefly from Smyrna and is very rich in coloring-matter.

The color solution of madder is best prepared by boiling the rasped wood in water and straining through cheese-cloth, making use of the clear solution for the dye-bath. The colors obtained on the various mordants are not as clear and bright as those produced from alizarin. The use of calcium acetate in the dye-bath serves the purpose of brightening the color; in case the water contains considerable lime this addition need not be made, but sufficient acetic acid should be added. Madder may be dyed in a single bath, using 5 per cent. of alum, 4 per cent. of tartar, 4 per cent. of calcium acetate, and 10 per cent. of madder; enter the material cold and slowly bring to the boil, and maintain at that temperature for one hour. This method is used only for the dyeing of light colors, as otherwise there would be considerable precipitation of coloring-matter in the dye-bath. A pale brownish drab stain on wool may be produced by boiling with a decoction of madder with the use of any mordant whatever. This method, in fact, has been used in practice. The colors produced with madder on either a chromium or an aluminium mordant may be considerably brightened by the addition of a small amount of tin crystals to the mordanting bath. In order to fully develop the coloring power of madder it is necessary that the temperature of the dye-bath be gradually and regularly elevated to the boiling point. The addition of a small amount of sumac (or other tannin extract) to the dye-bath serves to give better exhaustion of the coloring-matter.

3. **Archil.** — This coloring-matter is obtained from certain species of lichens, the principal varieties of which are *Roccella tinctoria*, *Roccella fuciformia*, and *Variolaria orcina*. The dye-stuff occurs in the form of a paste, and is prepared by treating the lichens to a process of oxidation in the presence of ammonia. The principal color-producing compounds existing in the lichens are *erythrin*, *lecanoric acid*, and *evernic acid*. The lichens are torn up into small fragments, placed in iron drums provided with stirrers, and mixed with a dilute solution of ammonia. The temperature is kept at about 100° F. for several days, during which time the mass undergoes a fermentation which causes the develop-

ment of the coloring-matter. When the latter ceases to increase (which is determined by making tests from time to time) the fermentation is stopped. The product so obtained is *archil paste*; *archil liquor* is prepared by removing the fibrous matter of the plant. *French purple* is a preparation of archil which is said to give faster shades than the ordinary product; it is made by treating the lichens with a dilute solution of ammonia, acidulating the resulting liquid with hydrochloric acid, which precipitates the coloring-matters. This precipitate is washed, dissolved in strong ammonia, and kept for about three weeks at a temperature of 160° F., during which time a fine purple color is developed; calcium chloride is added and a purple lake is precipitated. When used for dyeing this lake is mixed with an equal weight of oxalic acid and dissolved in water.

A solution of archil gives the following reactions:

*Acids*: Solution yellower.

*Alkalies*: Solution bluer.

*Lead acetate*: Crimson precipitate.

*Calcium chloride*: Red precipitate.

*Stannous chloride*: First redder, then yellower.

*Alum*: Solution redder.

*Basic alum*: Crimson-red precipitate.

The coloring principle of prepared archil is known as *orcein*.

Archil, or orchil, was formerly prepared by treating the lichens with water containing putrid urine, and at a subsequent stage with slaked lime. Archil is chiefly employed for the dyeing of carpet yarns.

Archil occurs in trade in three forms: (1) as a thick liquor called archil; (2) as a paste called *persis*; and (3) as a reddish brown or purple powder termed *cudbear*. The liquor varies in specific gravity from 8° to 20° Tw.; the paste is usually a 35 per cent. one, but is sometimes as low as 20 per cent. Archil is sometimes adulterated with other vegetable coloring-matters such as logwood, sapan, Brazil-wood, etc., and also with coal-tar dyes, especially Magenta. Pure cudbear is obtained from a



lichen known as *Lecanora tartarea*. It is prepared and used in exactly the same manner as archil, and gives the same colors; it also yields the same reactions. In fact, the two dyes commercially are not distinguished.

4. **Quercitron.** — This dyestuff is from the inner bark of a species of oak, the botanical name of which is *Quercus citrina* or *Quercus tinctoria*. It is found principally in Pennsylvania, Georgia, and the Carolinas. Its dyeing properties are due to two principles, *quercitrin*,  $C_{36}H_{88}O_{20}$ , and *quercetin*,  $C_{24}H_{36}O_{11}$ . The best varieties are shipped from Philadelphia, New York, and Baltimore; the Philadelphia variety being the most highly prized. It is principally used in calico printing for the production of compound shades in conjunction with mordants of aluminium, tin, chromium, and iron. It is also employed in woollen printing. In the dry condition, quercitron is of a yellow or buff color, being a mixture of the fibres with a fine powder of a bitter and astringent taste. The extract when freshly prepared is nearly transparent and of a dull orange-red color, which on standing deposits a yellow crystalline powder, and becomes turbid and considerably thicker. The extract is adulterated chiefly with molasses. In the form of extract it is known as *bark extract* and is usually sold at a density of  $51^{\circ}$  Tw.

*Patent bark* is prepared by boiling ground quercitron bark with dilute sulphuric acid, the product being washed and dried. Its chief use is as a substitute for flavine in wool dyeing.

*Flavine* is a very pure dry extract of the coloring-matter of quercitron bark. The best varieties contain a large proportion of quercetin, and yield yellow colors of great brightness. Flavine is still somewhat used in conjunction with cochineal for the production of bright yellowish scarlet colors.

The colors obtained from quercitron and flavine are dulled by prolonged boiling in the dye-bath owing to the presence of considerable tannin. The addition of glue solution is beneficial in this respect.

5. **Cutch.** — Cutch, or catechu, is the dried extract obtained from several Indian trees, a species of *Acacia*, the chief variety



being *Acacia catechu*. The principal varieties are Bombay, Bengal, and Gambier cutch. Bombay cutch is obtained from the fruit and wood of the *Arcea catechu*, a kind of palm. Bengal cutch is obtained from the twigs and unripe pods of the *Mimosa catechu*. The above two varieties are very similar in appearance, coming into commerce in the form of large blocks of a dark brown color, weighing from 30 to 40 pounds and packed in leaves. They are hard but brittle, and are imported from Java, Singapore, Peru, and the East Indies. Gambier cutch, called also cubical or yellow cutch, is obtained from the leaves of the *Uncaria gambier*, and occurs in trade in the form of small cubes. It is much more yellow in appearance than the two other varieties and is also much less soluble in cold water. It has a dull earthy fracture and is porous. The best variety is grown in Rhio, in the Isle of Brittany, and is imported from Singapore. Another variety of cutch is *kino*, or *gum kino*, which is obtained from the *Pterocarpus marsupium*. It has a reddish brown color and a highly lustrous fracture.

Cutch contains two principal coloring-matters, *catechin*,  $C_{19}H_{20}O_2$ , and *catechu-tannic acid*,  $C_{38}H_{36}O_{16}.H_2O$ . It is chiefly used in cotton dyeing and in calico printing for the production of brown shades or as a tannin mordant to be topped with basic or other colors. Good qualities of cutch should not contain more than 5 per cent. of ash on ignition, nor more than 12 per cent. of matter insoluble in alcohol. It is frequently adulterated with starch, dried blood, sand, and clay. Starch is detected by treating the sample with alcohol, filtering, and dissolving the residue in hot water, cooling, and testing for starch with an iodine solution. Pure cutch gives a decided green color with solutions of ferric salts, so the addition of other tannin matters may be recognized by the modified color given with ferric salts. Sand, clay, etc., are easily detected by making up a decoction of the sample and observing the amount of insoluble residue which remains, as pure samples should be almost entirely soluble in hot water. Blood may be detected in a similar way as starch, by treating the sample with alcohol, the residue being dried and

heated in a test-tube, when, if blood is present, ammonia and offensive odors are given off.

As already mentioned, cutch contains two coloring principles; the one is soluble in cold water and is termed catechu-tannic acid, or *mimotannic acid*; the other is nearly insoluble in cold water and is termed catechin or *catechuic acid*, a brown amorphous substance. Catechu-tannic acid may be obtained by boiling pulverized cutch with water, allowing the solution to stand for several days, when the catechin separates out and may be filtered off. The filtrate is evaporated to dryness and treated with alcohol to remove impurities. The product is a reddish brown powder soluble in water and alcohol, but not soluble on dry ether. With ferric salts it gives a grayish green precipitate, and gives no reaction with ferrous salts. Its aqueous solution is precipitated by gelatin, albumen, and sulphuric acid. Cutch may contain from 35 to 55 per cent. of catechu-tannic acid, according to its source. With alkalies catechu-tannic acid forms soluble salts, the solutions of which rapidly oxidize on exposure to the air and become of a reddish color.

Catechin forms that part of cutch insoluble in cold water. It is obtained in the pure state by taking the solid which separates out after boiling cutch with water and cooling; it is purified by redissolving in hot water, boiling with animal charcoal to decolorize it, filtering hot, and allowing to cool. These operations may have to be repeated several times. The product obtained is in the form of white silky crystalline needles, which are very slightly soluble in water. Catechin precipitates albumen, but not gelatin. When dissolved in concentrated sulphuric acid it gives a purplish colored solution. Though sometimes called catechuic acid, catechin has no acid properties, and is neutral to litmus. When dissolved in solutions of alkaline carbonates it rapidly absorbs oxygen from the air and becomes dark red in color; and on the addition of an acid dark red *rubinic acid* is precipitated. If caustic alkalies are used as the solvent, then a very dark brown, nearly black, precipitate of *japonic acid* is obtained under similar conditions. This same substance is formed when a decoction of

cutch is oxidized with potassium bichromate; and in fact, it is on this property that the dyeing powers of cutch depend.

Dyers utilize the coloring properties of both catechin and the catechu-tannic acid, but the calico printer requires chiefly the catechin. In general, cutch is used in cotton dyeing for the production of browns and as a tannin mordant; it is, however, somewhat used in woolen and silk dyeing, being employed in the latter chiefly as a weighting and mordanting agent in the production of blacks. It is also used for the dyeing and preserving of sails and fishing nets, as well as in medicine as an astringent, and also in the tanning of leather.

Cutch is best applied to cotton by boiling the goods in a decoction of the dyestuff and then allowing to stand for some time after which the cotton is taken out, squeezed, and worked in a hot bath containing potassium bichromate, which acts on the soluble catechin and catechu-tannic acid to produce insoluble japonic acid on the fibre. Some dyers enter the cotton into a hot bath of cutch, then work it for some hours without further application of heat, and treat it as before with chrome. It is possible to use bluestone instead of chrome, and if the color is developed by the former it appears much yellower and not quite so full in shade as the colors produced by the latter. If bluestone is used it is the better plan to add it directly to the bath containing the cutch, and afterwards to develop in the usual way with chrome. In the latter case, the shades are fuller and faster to light than would be the case if no bluestone were used. Copperas may also be added to the bath for the purpose of darkening the shade. It is advisable when dyeing very dark cutch browns to first work in a fairly weak bath, develop in the chrome and afterwards work in the cutch bath again, and again develop with chrome, and repeat this until the required depth of shade is obtained. By this means, darker, fuller, and more level shades may be obtained than by using very strong solutions of cutch. This method is especially applicable to the dyeing of warps and cotton pieces. It should be noted that the presence of copper in the color-lake appears to make it faster to light. Instead of using



the copper sulphate directly in the cutch bath, as is usually done, the cotton may be worked in a cold solution of the salt, either on coming out of the cutch bath or after being developed. Though chrome and bluestone are the chief metallic salts employed for fixing cutch in cotton dyeing, other salts may also be used. Aluminium salts give a yellowish brown color, while tin salts give a still yellower color; copperas gives a brownish gray. Cotton dyed with cutch has the property of being afterwards dyed with the basic and with alizarin (or natural) dyestuffs. In the former case it is the catechu-tannic acid, or the products formed from it by oxidation, that act as the mordant; in the latter case it is the chromium or copper fixed in the fibre which acts as the mordant. It is apparent, therefore, that if the tone of a cutch brown has to be altered this may be accomplished by any suitable dyestuff of the above groups. In the case of the coloring-matters requiring a metallic mordant, the dyestuff may be added directly to the cutch bath, when, of course, the color produced by it would be developed at the same time as the cutch. With the basic colors, however, it would be necessary to first dye the cutch brown and then top off in a separate bath with the basic color.

At the present time cutch is very little used as a dyestuff for wool, although for the production of certain brown shades it might be employed with advantage. The objections to the use of cutch are several: (a) The wool acquires a harsh feel; this might be remedied to a certain extent by using only the catechin, but this is too expensive. (b) The best and fastest shades are produced by the so-called "saddening" process; that is, first boiling the wool with the coloring matter and then fixing in a fresh bath with a solution of a metallic salt. As this process is not a very convenient one for dyeing to shade, it is easy to understand why cutch is not much used. The manner of dyeing wool with cutch is very similar to that for the dyeing of cotton, except that boiling solutions are used. Cutch may also be used on wool in conjunction with such dyes as barwood and camwood. By first mordanting with chrome or other metallic salt (bluestone or copperas), lighter shades are obtained than when the saddening method is



used. The colors obtained with chrome are fairly fast to light and milling, and by the addition of a little bluestone to the cutch bath these properties are increased.

In silk dyeing cutch is used for two purposes. One is for the dyeing of silk plush an imitation of sealskin; in which case the silk is dyed in a similar manner to cotton. The second is the use of cutch in black dyeing, when the method is to first mordant the silk with nitrate of iron, and dye with Prussian blue, after which the silk is worked in a strong decoction of cutch, or better, gambier, to which may be added a small amount of tin crystals. The silk absorbs a large percentage of catechin, and is then mordanted with pyrolignite or nitrate of iron and dyed in the usual manner. This method is used in the production of the so-called "Lyons" black, where it is desired to weight the silk about 10 per cent.

6. **Cochineal.** — This coloring-matter is derived from an animal source. It consists of the bodies of the female insects known as *Coccus cacti*; they are found in Mexico and Central America and other tropical and sub-tropical countries, and grow on certain kinds of cactus. At the proper time the insects are collected and killed by being steamed or dried in hot stoves; the former gives the *black cochineal* and the latter the *silver cochineal*.

The coloring principle of cochineal is *carminic acid*. The aqueous solution of cochineal yields the following reactions:

*Acids*: Yellowish color.

*Alkalies*: Violet color.

*Lime-water*: Violet precipitate.

*Alum*: Slowly forms red precipitate.

*Aluminium chloride*: Reddish violet precipitate.

*Stannous chloride*: Violet precipitate.

*Stannic chloride*: Bright scarlet color.

*Ferrous sulphate*: Violet gray precipitate.

*Copper sulphate*: Violet precipitate.

*Lead acetate*: Violet precipitate.

*Zinc sulphate*: Violet precipitate.

*Oxalic acid*: Red precipitate.

Cochineal was formerly very extensively employed for the production of bright scarlets and reds on wool; it is still used to some extent for this purpose, but has been largely replaced by the acid scarlets. The scarlet cloth for the English army, however, is still dyed with cochineal. In cotton dyeing cochineal has no application, though small quantities are used in printing. Cochineal gives the following colors with the different mordants:

*Chromium*: Purple.

*Aluminium*: Crimson.

*Iron*: Purple.

*Copper*: Claret.

*Tin*: Scarlet.

The principal colors are the crimson with alum and the scarlet with tin. Cochineal scarlet is faster to light than the acid scarlets; it is also quite fast to washing and fulling, but becomes a little bluer, though it does not bleed. The solution of the coloring-matter for dyeing is best prepared by boiling the powdered cochineal insects in water and straining the solution.

*Ammoniacal cochineal* is a preparation obtained by steeping ground cochineal in ammonia water for several days, three parts of ammonia being used for one part of cochineal. A chemical reaction takes place resulting in the formation of a carminamide from the carminic acid. The mixture is then heated to drive off the excess of ammonia, and hydrated aluminium oxide is added, and the heating continued until all of the ammonia is removed; then the mass is pressed into cakes. It is used for dyeing purple and crimson, and for rose reds in connection with ordinary cochineal. Its color is not as readily affected by acids as that of the other cochineal. It also gives a fine purple precipitate with oxychloride of tin.

A good quality of cochineal should not give more than one per cent. of ash on ignition. It is frequently adulterated with half exhausted cochineal which is made to resemble white or silver cochineal by drying and agitating with barium sulphate, white lead, etc. Black cochineal is also adulterated with black iron,

sand, graphite, and black oxide of manganese. These mineral adulterants are easily detected by powdering the sample and treating with water, when the mineral matters will in most cases fall to the bottom. Occasionally, adulteration is practiced by adding extract of Brazil-wood. This may be detected by treating the sample with water, adding an excess of lime-water, which completely precipitates the coloring-matters of the cochineal, while if Brazil-wood is present the filtered liquid will have a purple or violet color. The value of different samples of cochineal is best estimated by dissolving a given weight of the powdered samples in water and observing the amount of standard alum solution necessary to completely precipitate the coloring-matters. A more accurate method, perhaps, is to conduct a series of comparative dye-tests using test-skeins of woolen yarn previously mordanted with tin or alumina.

*Cochineal carmine*, or *carmine lake*, is a brilliant red pigment produced by precipitating a decoction of cochineal with alumina. Its manufacture, however, is still maintained as a trade secret. It contains a large amount of alumina and lime, combined with a certain amount of nitrogenous matter, which seems to be essential to its formation. It is chiefly used by paper stainers and calico printers. Cochineal carmine is liable to be adulterated with starch, china clay, vermilion, and various pigment colors. These additions may be detected by treating the sample with dilute ammonia water, which will readily and completely dissolve pure samples, while if any of the above named substances are present they will be left as insoluble matters. The ash should be under 10 per cent., and the water should not be over 20 per cent. The ash should be examined for tin, which if present in any considerable amount indicates the presence of Biebrich Scarlet lake, which closely resembles cochineal carmine in many of its properties, and is somewhat difficult to detect in small quantities.

Besides the usual two-bath process of dyeing cochineal a one-bath method may also be used, as follows: The dye-bath is prepared with 6 per cent. of oxalic acid, 6 per cent. of stannous chloride, and 20 per cent. of cochineal. The oxalic acid should be added



before the tin crystals, otherwise a precipitate of stannous oxy-chloride will occur which will cause loss of coloring-matter. A deficiency of tin causes the color to be dull and bluer, while an excess of tin gives a paler scarlet. The one-bath method gives yellower and more brilliant shades than the two-bath process, though more cochineal is required. The presence of iron or copper in the dye-vat should be avoided, otherwise the scarlet will be much dulled. To obviate this defect arising from the use of copper steam-pipes in the dye-vat a piece of clean tin should be placed in the bath; this prevents the copper from being dissolved. For the production of very yellow tones of scarlet it is necessary to use some suitable yellow dyestuff in connection with cochineal. Flavine was generally employed for the purpose.

#### SAMPLES.

- 387. Wool dyed with fustic on chrome mordant.
- 388. Wool dyed with fustic on tin mordant.
- 389. Wool dyed with fustic on iron mordant.
- 390. Wool dyed with madder on chrome mordant.
- 391. Wool dyed with madder on aluminium mordant.
- 392. Wool dyed with madder on tin mordant.
- 393. Wool dyed with archil in neutral bath.
- 394. Wool dyed with archil in acid bath.
- 395. Wool dyed with quercitron on chrome mordant.
- 396. Wool dyed with flavine on tin mordant.
- 397. Cotton dyed with cutch.
- 398. Showing effect of bluestone in dyeing cutch.
- 399. Wool dyed with cochineal on tin mordant.
- 400. Wool dyed with cochineal on aluminium mordant.

#### QUIZ 20.

600. Name the principal minor natural dyestuffs. What colors do they yield, and on what fibres are they chiefly dyed?

601. Describe the method of dyeing wool with fustic. What is the effect of the use of different mordants? How does the fastness of fustic to washing and light compare with that of alizarin yellow?

602. From what tree is fustic obtained? What is its botanical name? In what countries does it grow?

603. What colors does fustic yield with the different mordants? In what forms may it be employed? In the dyeing of what colors is it principally used?

604. What coloring-matters are present in fustic? What differences do they exhibit?

605. Describe the principal reactions given by a solution of fustic.

606. In what forms does fustic occur in trade? With what is fustic extract chiefly adulterated? How may fustic extracts best be tested?

607. What is the effect of boiling the dye-bath too long when using fustic? Explain the reason and state how the defect may be prevented.

608. What is "young fustic"? How does it compare with ordinary fustic? Is it still in general use?

609. What is madder? Give the general method of applying it to wool. What mordants may be used, and how do the different mordants affect the color?

610. From what is madder obtained? In what countries was it largely cultivated? What dyestuff has now replaced madder?

611. What are the principal coloring-matters contained in madder? Which is the most important constituent? In what form do the coloring-matters exist in the madder root, and how are they developed?

612. How may alizarin be obtained from madder? What are its principal reactions? How may it be converted into anthracene?

613. In what forms was madder employed in dyeing? How is garancin prepared, and how does it compare with madder? What are "garanceux" and "flowers of madder"?

614. What colors does madder yield with the different metallic mordants? In what dyeing process is madder still somewhat employed?

615. What are the chief varieties of madder? What is "crop madder" and "muelle madder"?

616. How is the color solution of madder best prepared? How do the colors obtained with madder compare with those from alizarin?

617. How may madder be dyed in a single bath? Why cannot this method be used for dyeing heavy shades?

618. How may the color obtained on an aluminium mordant with madder be brightened? What is the effect of adding sumac to the dye-bath?

619. From what is archil obtained? How is it prepared? What is the difference between archil paste and liquor?

620. How is French purple prepared? How is it used in dyeing? To what other dyestuff is archil closely related?

621. What class of dye may archil be considered with respect to wool? What color does it give? What is the general fastness of the color?

622. Give the method of dyeing archil on wool. What is the effect of adding acid to the bath?

623. What is the chief use of archil? Is it used in cotton dyeing? How may it be dyed on silk?

624. What are the general reactions of a solution of archil? By what other name is archil known? For what class of yarns is archil principally used?

625. In what forms does archil occur in trade? How does cudbear compare with archil?

626. What color does quercitron give? From what is it obtained? What is flavine?

627. Give the method for dyeing quercitron. What is the difference in its color on a chrome and a tin mordant?

628. In what branch of dyeing are quercitron and flavine still somewhat used? What is the general fastness of the color?

629. What is patent bark and how is it prepared? What is the effect of prolonged boiling in the dyeing of quercitron and flavine?

630. What color does cutch yield? What other use than as a dyestuff does it possess? On what fibres is cutch dyed?

631. Give the general method of dyeing cutch on cotton. How does the addition of bluestone affect the dyeing?

632. From what is cutch obtained? How does gambier differ from Bombay cutch? What coloring-matters does cutch contain? What is formed when cutch is oxidized with chrome?

633. What would be the effect on the color of cutch of adding copperas in the dyeing? For what purposes is cutch used in the dyeing of silk?

634. Of what does cochineal consist? What is the difference between "black" and "silver" cochineal? What colors does cochineal give on mordants of tin and of aluminium?

635. Give the method of dyeing cochineal scarlet on wool. What is the general fastness of the color?

636. What is ammoniacal cochineal? How is it prepared? What is cochineal carmine?

637. What adulterations are liable to be found in samples of cochineal, and how would you conduct tests for their detection?

638. Give the method of dyeing cochineal by a one-bath process. The presence of what metals should be avoided in dyeing cochineal? How are very yellow tones of cochineal scarlet obtained?

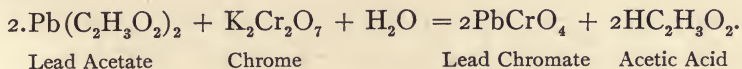


## SECTION XXI.

### THE MINERAL DYESTUFFS.

**Experiment 125. Chrome Yellow on Cotton.** — Steep a test-skein of cotton yarn for 30 minutes in a cold bath consisting of a 5 per cent. solution (5 grams per 100 cc.) of lead acetate. Squeeze evenly, and pass into a second bath consisting of a 1 per cent. solution (1 gram per 100 cc.) of chrome; work cold for 30 minutes. Squeeze and wash in fresh water, then soften by working in a dilute solution of a cotton softener or glycerin and soap. Finally squeeze and dry (401). In order to obtain heavier colors the alternate passages through the baths of lead acetate and chrome may be repeated several times. In place of using the ordinary acetate of lead (sugar of lead) the subacetate is preferred by some. This is prepared by boiling together 10 parts of lead acetate and 6 parts of litharge (lead oxide) with 40 parts of water; filter, and use the liquor so obtained, diluting in accordance with the depth of color desired.

Chrome yellow is formed in accordance with the following reactions:



Chrome yellow may be applied to wool, silk, or any other fibre in the same manner as above described for cotton, but it is seldom, if ever, used in these fibres. In the dyeing of chrome yellow it is necessary to first apply the lead salt and then the chrome; if the reverse procedure is followed the pigment will be precipitated on the outside of the fibres in a loosely adherent condition, and will not be fast to washing. In dyeing heavy shades, in order to get the most even results and the fastest color, it is best not to use more concentrated solutions but to

give the cotton several dips successively in the two solutions until the desired depth of shade is obtained. To obtain the purest shades of yellow, it is best to have the chrome bath slightly acid, for if the latter becomes at all alkaline the resulting pigment will acquire an orange tone. On this account it is better to employ the bichromate of potash rather than the neutral chromate. Chrome yellow, though fast to light, washing, and acid, is quite sensitive to the action of sulphuretted hydrogen, turning dark, owing to the formation of black lead sulphide. As the air of cities, especially in the vicinity of factories, and the air of houses heated by burning coal, always contain more or less sulphuretted hydrogen, this accounts for the gradual darkening of chrome yellow on exposure. This discoloration can be prevented to a considerable extent by incorporating with the lead salt a salt of zinc or cadmium, the sulphide of the former being white and that of the latter yellow in color. In order to show this action, add to the bath of lead acetate used in the above experiment 1 per cent. of cadmium nitrate; then dye a second skein of cotton in the same manner as the previous one (402). Take small samples of the two skeins and place them in a bottle, the air of which contains a minute quantity of sulphuretted hydrogen. After some time it will be found that the first sample, dyed with the lead salt alone, has become perceptibly darkened, whereas the second sample, containing the addition of cadmium salt, is not altered. Though unaffected by acids, chrome yellow is changed to an orange by the action of alkalies; even lime-water will serve this purpose. The orange color is due to the formation of a basic compound of lead chromate. To illustrate this action, take a small sample from the skein dyed with chrome yellow and boil it in a weak solution of soda ash; then wash and dry. It will be found to have changed to an orange color. Treatment with acid will in turn destroy the orange tone and restore the original yellow color; this may be shown by steeping the sample above tested in a dilute solution of sulphuric acid, when the color of the original chrome yellow will again be formed. By the action of strong caustic alkalies,

chrome yellow may be completely discharged or dissolved from the fibre, as may be shown by taking a small sample from the skein dyed with this color and boiling it in a solution of caustic soda, when it will be found to become rapidly decolorized. This reaction is very useful in printing, as by its means discharge effects may be obtained.

**Experiment 126. Chrome Orange on Cotton.** — As already indicated in the previous experiment, this color may be obtained by forming the basic chromate of lead in the fibre by the use of lead chromate and an alkali. Proceed as follows: Work a test-skein of cotton yarn for 30 minutes in a cold bath consisting of a 5 per cent. solution of lead acetate; squeeze, and pass into a second bath consisting of a 1 per cent. solution of chrome and a small quantity of caustic soda. Enter cold and gradually raise to the boil for a few minutes. Wash in a warm dilute soap bath (403).

A modification of the above method is to use the basic acetate of lead prepared in the manner prescribed in the previous experiment from lead acetate and litharge.

The chrome orange obtained as above indicated may be brightened somewhat by working in a boiling bath containing lime. Dye a second skein of cotton in a manner similar to the first, repeating the treatment in the two baths three times. Squeeze and wash, then work for 15 minutes at the boil in a bath containing 10 per cent. of lime (quicklime). Finally wash and soap as before (404).

By a stronger or weaker treatment with alkali, chrome orange may be made to vary in shade from a bright yellowish orange to a scarlet red. The remarks made under chrome yellow as to its fastness and reactions with various agents are also applicable to chrome orange.

Both chrome yellow and chrome orange are poisonous substances, and may give rise to cases of poisoning among operatives handling cotton dyed in this manner, or even to wearers of such fabrics. These dyes may be tested for on the fibre by boiling a sample in caustic soda solution and then adding a few drops of ammonium sulphide solution, when a black precipitate of lead sulphide will be formed.



**Experiment 127. Iron Buff on Cotton.** — This color is produced by precipitating a hydrated oxide of iron ( $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ) in the fibre. Proceed as follows: Work a test-skein of cotton yarn for 30 minutes in a cold bath consisting of a 5 per cent. solution of copperas (ferrous sulphate,  $\text{FeSO}_4$ ). Squeeze, and pass into a bath containing 5 per cent. on the weight of the cotton of soda ash; work for 15 minutes at  $180^\circ\text{F}$ . Wash and pass through a warm dilute soap bath (405). The reaction takes place as follows:



The oxidation of the iron from the ferrous to the ferric condition is effected by the atmospheric oxygen. By repeating the treatment with the two baths several times heavier shades of brown may be obtained. Instead of using copperas a solution of "nitrate of iron" (basic ferric sulphate) may be substituted, or a solution of ferric chloride. Another method of procedure is as follows: Work a skein of cotton as above in the same bath of copperas; squeeze, and pass through a cold weak solution of chloride of lime containing a little caustic soda for 10 minutes. Squeeze, and repeat the passage through the two baths twice. This should give quite a heavy shade of brown (406). Wash well, and soap as before. The chloride of lime oxidizes the ferrous salt very rapidly to the ferric condition; it also forms a certain amount of oxy-cellulose with the cotton which takes up the iron compound more energetically than the unmodified cotton. Another method of producing iron buff on cotton is to impregnate the material with the solution of the iron salt as before, then to pass it through a bath containing milk of lime, after which it is squeezed and exposed to the air over-night. This latter operation is termed "aging." The light brown shade obtained with iron oxide is also known as *nanking* and *chamois*. The brown color of the natural Nanking cotton is no doubt due to its containing oxide of iron. Iron buff on cotton is exceedingly fast to light, washing and alkalies, and also to exposure; it is decolorized, however, with acids, which may be shown by steep-

ing a small sample of the dyed skein in a warm dilute solution of hydrochloric acid. In calico printing iron buff may be discharged white with citric acid or with a solution of stannous chloride in hydrochloric acid.

As iron oxide forms a good mordant with the alizarin and natural dyestuffs, iron buff on cotton may be topped off with these dyestuffs and quite an extensive variety of shades produced thereby. In order to illustrate this procedure, dye three skeins of cotton a light shade of iron buff in the manner above indicated. Top off the first one in a bath containing 2 per cent. Alizarin Red (407), the second one with 2 per cent. Alizarin Blue (408), and the third one with 5 per cent. fustic extract (solid) (409). Enter at a low temperature and gradually raise to the boil. Wash and soap in the manner before described.

**Experiment 128. Iron Gray on Cotton.** — This color is obtained by precipitating tannate of iron within the fibre. Proceed as follows: Work a skein of cotton for 30 minutes in a cold bath of nitrate of iron at 2° Tw.; squeeze, and pass into a bath containing 5 per cent. of tannic acid; work cold for 15 minutes. Wash and soap in the usual manner (410). Deeper shades of gray and slate may be obtained by repeating the treatment several times. The operations may also be reversed and the treatment with the tannic acid may take place first, as in the usual manner of mordanting cotton for the purpose of dyeing heavy colors with the basic dyes. Besides tannic acid itself the various natural tannins may be employed, such as sumac, cutch, chestnut extract, etc., in which cases the resulting color will be modified by the addition of the natural color of the tannin. By using rather concentrated baths and repeating the operations several times, cotton may be dyed black by this method. In fact, before the introduction of logwood, this was the chief method for the dyeing of black on cotton. Iron gray on cotton is quite fast to light and washing; on long exposure it turns rusty, owing to the gradual formation of iron oxide; it also turns brown on treatment with alkalis for the same reason. Like iron buff it is also decolorized by the action of acids.

**Experiment 129. Manganese Brown on Cotton.** — This color is also known as "bistre," and is formed by precipitating an oxide of manganese in the fibre. Proceed as follows: Work a skein of cotton for 30 minutes in a cold bath consisting of a 5 per cent. solution of manganese chloride; squeeze and pass into a cold bath containing 10 per cent. of caustic soda; work for 15 minutes; wash in fresh water, and then pass into a dilute bath of chloride of lime (about 1° Tw.); finally wash well and soap in the usual manner (411). In the treatment with caustic soda there is precipitated in the fibre a hydrate of manganese; a dilute bath of soda ash may also be used for the same purpose. The final treatment with chloride of lime is for the purpose of oxidizing the compound to the higher oxide of manganese. The resulting compound is probably  $Mn_2O_3$  and consists of a mixture of manganese dioxide,  $MnO_2$ , and manganous oxide,  $MnO$ . Bistre was formerly a very important color for cotton and extensively used both in dyeing and printing. It is very fast to light, washing, and alkalis; it is also fast to dilute acids, but strong acids decolorize it, as also do reducing agents. Bistre may also be dyed on cotton by passing the material saturated with the solution of manganese chloride into a bath containing a mixture of caustic soda and chloride of lime, an operation which then dispenses with the use of a third bath. The use of soda ash in place of the caustic soda cannot be recommended, as the precipitate produced contains manganese carbonate, which is not as readily oxidized as the hydrate. The final dyeing is also apt to come out rather uneven. Bistre can also be produced on cotton by the use of potassium permanganate, as may be shown in the following manner: Work a skein of cotton for 15 minutes in a cold bath containing 2 per cent. of potassium permanganate. The cotton will be found to turn brown rapidly on exposure to the air; squeeze, wash, and soap in the usual manner (412). Manganese brown is decolorized by treatment with either hydrogen peroxide or sulphurous acid; wherein it differs from the brown obtained from iron oxide. In order to show this behavior, take a small sample each of iron buff and bistre and



steep them for several hours in a solution of hydrogen peroxide; also steep two similar samples in an acidified solution of sodium bisulphite. It will be found that the samples of bistre are more or less completely decolorized, while the samples of iron buff are not much altered. Bistre may be employed as the basis for the production of aniline black on cotton, proceeding as follows: Take a skein of cotton dyed a full shade of brown with bistre in the manner above described, and work it in a cold bath containing 10 per cent. of aniline salt; then gradually bring to the boil. Squeeze, wash thoroughly, and soap in the usual manner (413). This black is very fast to washing. By using paraphenylene-diamine or beta-naphthylamine, a very good shade of brown may be obtained which does not differ much in color from the original bistre, but it is fast to acids. With alpha-naphthylamine a plum color is produced. Cotton cloth dyed with bistre has the property when subsequently dyed in the indigo vat of taking up a greater amount of indigo and fixing it faster to washing than ordinary cotton. Bistre is sometimes used in dyeing of mohair plush in order to give a fabric in imitation of a natural fur pelt, the cotton back being dyed with cutch brown in the yarn, while the mohair pile is woven from undyed yarn. The plush is then treated with a solution of potassium permanganate, which rapidly dyes the mohair brown and also colors the cotton back a fuller shade. As soon as the desired shade is obtained the material is washed and dried. Then by the use of rotating brushes a suitably thickened solution of sodium bisulphite is applied to the ends of the mohair pile, which causes the brown color to become discharged, and thereby imitate more closely the appearance of the natural pelt. In order to show the use of bistre on woolen material, take a skein of wool and pass it through a cold bath containing 2 per cent. of potassium permanganate; work for 15 minutes; then squeeze and wash well (414).

According to certain chemists, the irregularity which sometimes arises in manganese brown is due to the physical condition of the precipitate itself. In order to overcome such defects, it

has been recommended, after impregnating the cotton with the manganese salt, to pass it through a bath containing ammonia and potassium bichromate, whereby a rather unstable manganese chromate is precipitated in the fibre; this gradually decomposes, and the chromic acid liberated reacts with manganous hydrate, forming the higher oxide of manganese. The oxidation is completed by passing the cotton through a dilute bath of bleaching powder.

**Experiment 130. Chrome Green on Cotton.**— A pale dull shade of green can be obtained on cotton by precipitating on the fibre oxide of chromium,  $\text{Cr}_2\text{O}_3$ . Proceed as follows: Work a test-skein of cotton for 30 minutes in a cold bath consisting of a 10 per cent. solution of chrome alum; squeeze, and pass into a bath containing 10 per cent. of soda ash; enter cold and gradually bring to the boil. Wash well and soap in the usual manner (415). By repeating the operations several times fuller shades may be obtained. Chromium oxide gives a sea-green color on cotton which is exceedingly fast to light, washing, and alkalis; it is also fast to exposure, but is decolorized by the action of acids. The color of chrome green may be brightened somewhat by passing the dyed cotton through a bath of dilute copper sulphate (the bath should be very dilute and warm). Chrome green is seldom used at the present time as a self color on cotton, but it has had extensive use in conjunction with iron buff for the production of the so-called *khaki* color with which the service uniform of the army is dyed. In order to obtain this khaki color proceed as follows: Work a test-skein of cotton in a cold bath consisting of a 5 per cent. solution of ferric chloride with a 5 per cent. solution of chrome alum, then pass into a bath containing 10 per cent. of soda ash; enter cold and gradually bring to the boil. Wash well, and soap as usual (416). By varying the relative amounts of iron and chromium salts, or by the addition of a small amount of manganese salt, the shade of this khaki color may be varied in order to obtain any tone desired.

**Experiment 131. Prussian Blue on Cotton or Wool.**— The production of this color depends on the precipitation of a ferro-

cyanide of iron within the fibre. On cotton it is dyed as follows: Work a test-skein of cotton in a boiling bath of nitrate of iron ( $32^{\circ}$  Tw.) also containing 5 per cent. of stannous chloride; steep for 30 minutes, squeeze, and pass into a bath containing 10 per cent. of potassium ferrocyanide (yellow prussiate of potash); work warm for 15 minutes; then pass back into the bath of nitrate of iron again; finally squeeze, wash, and soften in a soap bath (417). Heavier shades may be obtained by repeating these operations several times; the cotton, however, should always be worked last in the bath of nitrate of iron in order to prevent the formation of a soluble variety of Prussian blue. For dyeing wool proceed as follows: Work a test-skein of wool in a bath containing 10 per cent. of potassium ferricyanide (red prussiate of potash), 20 per cent. of sulphuric acid, and 1 per cent. of stannous chloride; enter cold and gradually raise to the boil, when the wool will turn green and finally become blue. After boiling for 10 minutes, lift, and add 1 per cent. more of stannous chloride, and work for 15 minutes longer. Finally wash well in fresh water (418). The depth of shade may be varied by employing greater or less amounts of potassium ferricyanide. If the blue color does not develop properly a few drops of nitric acid may be added to the bath for the purpose of accelerating the oxidation. Another method of dyeing this color on wool is to use 15 to 20 per cent. of potassium ferrocyanide with the addition of a small amount of alum and tartar to the bath.

Prussian blue also goes by the name of Berlin blue; it was formerly a very important color, both for cotton and wool, and is even still used to a considerable extent, especially in printing. Before the introduction of Alizarin Blue it was extensively employed for the dyeing of army uniforms. Prussian blue appears to be a complicated cyanogen compound of iron, the exact tone of which varies considerably with the manner of its production. Though not now employed as a self color in dyeing of silk, Prussian blue, however, is still used as a bottom color in the dyeing of weighted black on this fibre. Prussian blue is fast to light, washing, and exposure; it is also fast to dilute acids,



but is dissolved by stronger acids, also by a concentrated solution of oxalic acid. With caustic alkali it is decomposed into potassium ferrocyanide and brown oxide of iron. This latter reaction is still used for discharge work in printing. The action of stannous chloride in the dyeing of Prussian blue is to brighten and give a reddish tone to the shade, probably due to the formation of a tin ferrocyanide. A bright green color on cotton may be produced by the combined and simultaneous use of Prussian blue and chrome yellow in the following manner: Work a test-skein of cotton in a cold bath containing 10 per cent. of ferrous acetate and 10 per cent. of lead acetate for 30 minutes; squeeze, and pass into a bath containing 5 per cent. of potassium ferrocyanide and 2 per cent. of potassium bichromate. Squeeze, wash well, and soap as usual (419).

Boiling soap solutions decompose Prussian blue, leaving the brown oxide of iron on the fibre. On prolonged exposure to sunlight, the color becomes somewhat lighter, but the original tone is restored on being kept in the dark for some time.

The theory of the application of Prussian blue to wool is that when a mineral acid is added to a solution of potassium ferricyanide, the corresponding hydro-ferricyanic acid is liberated; this under the influence of heat and oxidation is decomposed with the precipitation of Prussian blue. If nitric acid is employed in the bath, the shade of blue is somewhat greener than when the other mineral acids are used. Yellow prussiate of potash may be used instead of the red, in which case it was the custom of dyers to use a mixture of the three mineral acids, under the name of "royal blue spirits," or simply "blue spirits." Nitric acid is the best acid to employ in connection with potassium ferrocyanide on account of its oxidizing action. The stannous chloride was formerly used by the dyer in the form of a solution known as "muriate of tin" or "finishing blue spirits." The solution in this form often contained sulphuric and oxalic acids.

## NOTES.

1. **General.** — There are a few mineral compounds which are capable of being used for the dyeing of textile fabrics. Though formerly of considerable importance, this class of colors is now nearly obsolete in dyeing. They differ very radically from the vegetable and coal-tar colors in that they are of mineral nature and are not organic bodies. As a rule, they are exceedingly fast to light, and are also very fast to washing. The general method of dyeing these colors is to impregnate the fibre with a solution of some metallic salt, and subsequently to treat it with a solution of another compound capable of yielding a colored precipitate with the metal already present. Lead salts, for instance, when added to potassium bichromate give a bright yellow precipitate of chrome yellow (lead chromate); if this precipitation is produced within the fibre itself, then the latter will become dyed with the chrome yellow. Cotton is the fibre mostly used for the application of the mineral colors, the only color which is applied to wool being Prussian blue. All the mineral dyes make the fabric more or less harsh and stiff; this may be remedied somewhat by after-soaping, or by using a cotton softener of oil, but it can never be removed entirely. Many of the mineral compounds used in the preparation of these colors are of a poisonous nature, which is a great drawback to their use; lead, copper, arsenic, mercury, and antimony compounds are all poisonous. As many of the metals forming the basis of these colors also serve as mordants with alizarin and many acid dyes, the colors obtained with the metallic pigments may be shaded and modified by the use of suitable coal-tar dyestuffs. Loose cotton is seldom dyed with the mineral colors, as it then becomes difficult to card and spin. The mineral colors, though now but little used in actual dyeing, are still employed rather extensively in calico-printing. In the latter they are used in connection with albumen in the color pastes, and this on steaming becomes coagulated and rendered insoluble, and at the same time serves to fix the color on the cloth.

The mineral colors differ in the principle of their dyeing from that of the coal-tar dyes in that they are strictly of a pigment character. There is no combination between the coloring-matter and the fibre itself; there is only a uniform precipitation of the finely divided insoluble pigment throughout the cells of the fibre, caused by the chemical double decomposition between the two soluble salts employed. The metallic salt is absorbed by the fibre from its solution by osmosis into the cells of the latter; as the osmotic action is comparatively slow, in order that the final dyeing be thoroughly penetrated, it is advisable to allow the cotton to steep in the solution of the metallic salt for a considerable time, and before being entered in the bath the yarn or cloth should be thoroughly wetted out, else the fibre will not become completely impregnated with the salt. After thorough saturation the goods may even be rinsed in fresh water without fear of washing out the metallic salt held in the pores or cells of the fibre; in fact, a moderate rinsing may be considered beneficial, as it serves to remove the excess of solution adhering to the outside of the fibres and between the interstices of the fibres themselves, as this is not removed by a simple squeezing or wringing. This portion of the metallic salt solution not held osmotically by the fibre would come off to a certain extent in the succeeding bath wherein the pigment is formed, thus causing an unnecessary consumption of chemicals, and the contamination of the second bath with a precipitate. There would also be formed a loosely adherent precipitate of pigment in the interstices between the fibres, which would not prove fast to washing or rubbing, and in the case of yarn would also dust off in the handling thereof, besides adding considerably and unnecessarily to the harshness of the cotton. The action of the second or precipitating solution is also by osmosis. Taking the formation of chrome yellow as an example, the solution of potassium bichromate gradually passes by osmosis into the pores of the fibre, where it comes in contact with the lead acetate already present; the insoluble chromate of lead separates out, and thus is held securely in the pores of the fibre, while the second member



of the reaction, the potassium acetate, being a soluble salt, passes back into the bath again by osmosis.

Most of the mineral colors are very cheap in their application, but it is rather difficult to dye them to a matched shade. Their exceptional fastness to light and washing is their principal advantage.

**2. The Minor Pigment Colors.** — There are a number of metallic pigment colors which may be produced in the fibre besides the ones which have been mentioned in the foregoing pages. They are, however, of only theoretical value and possess no practical importance to the dyer. A brown color may be dyed on wool by working it in a bath containing lead acetate and lime; the sulphur present in the wool combines to form lead sulphide. A gray color on cotton may be produced by working the latter in a bath containing mercury nitrate, squeezing, and passing through a bath containing sodium sulphide. Cotton may be dyed with red oxide of lead by steeping in a bath of lead acetate and then passing through a bath containing a mixture of caustic soda and chloride of lime. A blue color on cotton may be obtained by working in a bath containing ammonium molybdate and developing in a bath containing stannous chloride and hydrochloric acid. A yellow color on cotton or wool may be obtained by the use of titanium salts (see the application of these salts in mordanting). Cadmium yellow may be precipitated in cotton by first steeping in a solution of cadmium nitrate or chloride and passing through a bath containing sodium sulphide. Scheele's green may be dyed by first steeping the material in a solution of copper sulphate, then passing through a bath of caustic soda, whereby copper hydrate is formed, and finally treating with a solution of arsenious acid, resulting in the formation of green copper arsenite. Another green may be made in the fibre by steeping in a solution of chrome alum, passing through a bath of caustic soda and finally through a bath of sodium arsenite. Both of these green colors are very poisonous.

**SAMPLES.**

- 401. Chrome yellow on cotton.
- 402. Chrome yellow with cadmium salt.
- 403. Chrome orange on cotton.
- 404. Chrome orange brightened with lime.
- 405. Iron buff on cotton.
- 406. Iron buff oxidized with chloride of lime.
- 407. Iron buff topped with Alizarin Red.
- 408. Iron buff topped with Alizarin Blue.
- 409. Iron buff topped with fustic.
- 410. Iron gray on cotton.
- 411. Manganese brown on cotton.
- 412. Bistre dyed with potassium permanganate.
- 413. Aniline Black dyed on bistre.
- 414. Bistre dyed on wool.
- 415. Chrome green on cotton.
- 416. Khaki dyed on cotton.
- 417. Prussian blue on cotton.
- 418. Prussian blue on wool.
- 419. Green on cotton with Prussian blue and chrome yellow.

**QUIZ 21.**

639. Of what does chrome yellow consist? How is it applied to cotton? How are heavier shades obtained?

640. What is sugar of lead? How is sub-acetate of lead prepared? What chemical reaction takes place in the dyeing of chrome yellow?

641. On what fibres is chrome yellow dyed? Why is it necessary to use the bath of lead salt first? What is the effect on the color if the chrome bath becomes alkaline?

642. What is the general fastness of chrome yellow? Why is it not fast to exposure in city air? Explain the chemical reaction occurring.

643. How may the discoloration of chrome yellow be prevented? What is the effect of alkalis on chrome yellow? Of strong caustic soda?

644. How is chrome orange dyed on cotton? Of what does this color consist?

645. How may chrome orange be brightened? Through what range of color may chrome orange be produced? What is the general fastness of chrome orange?

646. What may be said regarding the poisonous character of the chrome colors? How may they be tested for on the fibre?

647. Of what does iron buff consist? How is it applied to cotton? Give the chemical reaction which takes place.

648. Give the method of dyeing iron buff by using chloride of lime. Explain the action of the latter.

649. By what names is the light shade of iron buff known? What is the general fastness of iron buff? How may it be discharged?

650. What dyes may be topped on iron buff as mordant? What is the general character of the colors obtained?

651. Of what does iron gray consist? How is it applied to cotton? What is its general fastness?

652. Of what does manganese brown consist? How is it applied to cotton? By what other name is it known?

653. What is the general fastness of bistre? How may bistre be dyed with potassium permanganate?

654. How may bistre be discharged? How is Aniline Black dyed on bistre? How may bistre be made fast to acid?

655. Of what use is bistre in indigo dyeing? How is bistre employed in imitating fur pelts? How may bistre be dyed on wool?

656. Of what does chrome green consist? What color does it give? How is it dyed on cotton? What is its general fastness?

657. What is "khaki"? How is it dyed? What is its fastness? How may the tone of the color be varied?

658. Of what does Prussian blue consist? How is it dyed on cotton? By what other name is potassium ferrocyanide known?

659. How is Prussian blue dyed on wool? How may the blue color be developed more rapidly?

660. By what other name is Prussian blue known? What dyestuff has replaced Prussian blue?

661. For what is Prussian blue used in silk dyeing? What is the general fastness of Prussian blue? How may it be discharged?

662. What is the function of stannous chloride in dyeing Prussian blue? What is the effect of boiling soap solutions on Prussian blue?

663. What is the theory of the dyeing of Prussian blue on wool? What is "blue spirits"? "Finishing blue spirits"?

664. What is the relative importance of the mineral dyes at present? How does their fastness to light, etc., compare with coal-tar dyes?

665. What is the general theory of dyeing mineral colors? In what branches of dyeing are the mineral colors still used?

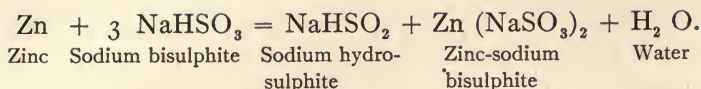
666. Name some of the minor pigment colors which may be dyed on cotton.



## SECTION XXII.

### THE VAT DYES.

**Experiment 132. Preparation of Indigo Solution.** — Mix 75 grams of indigo paste (20 per cent.) with 40 cc. of hot water, and 90 cc. of caustic soda solution (42° Tw.) and 50 grams of hydrosulphite powder (Blankit T) dissolved in 200 cc. of water. Stir gently and keep the temperature at about 110° F. In a short time the liquor should be of a clear amber-yellow color with a film of blue on the top. The liquid now contains indigo-white and serves as a stock indigo solution for the preparation of the dye-vat. The hydrosulphite powder (Blankit T) is a formaldehyde compound of sodium hydrosulphite and is a fairly stable body compared with most hydrosulphite derivatives. It is a strong reducing agent and converts the indigo-blue into indigo-white. In place of this prepared form of hydrosulphite the solution of sodium hydrosulphite itself may be used, in which case it may be prepared as follows: 130 grams of zinc dust are made into a paste with 55 cc. of water, which is then mixed with 1000 cc. of sodium bisulphite solution of 72° Tw. As the mixture is liable to become heated, the temperature should be kept below 100° F. by the addition of ice or cold water; when the chemical action has ceased, dilute to 2 litres and allow to stand for one hour. Then stir in 200 cc. of a 20 per cent. milk-of-lime, cold, and allow to stand for 2 hours. This causes the precipitation of all the zinc as zinc hydrate. The liquor is then strained to free it from sediment, and preserved in a closed bottle. The hydrosulphite solution thus prepared, if kept in a cool place, will last for several weeks, and its keeping quality will be enhanced by the addition of a small quantity of caustic soda solution. When zinc dust reacts with a solution of sodium bisulphite the following chemical change takes place:



Sodium hydrosulphite is a strong reducing agent, being itself oxidized finally to sodium bisulphate,  $\text{NaHSO}_4$ . Its use forms a very convenient means for the preparation of dyeing solutions of indigo as well as the other vat dyes. The sodium hydrosulphite solution is usually prepared by the dyer as a stock solution, and used as occasion requires for the reduction of the dyestuff to be added to the vat.

**Experiment 133. Dyeing Indigo with Hydrosulphite Vat.** — Prepare the dye-vat as follows: to one litre of water ( $70^\circ \text{F.}$ ) add 2 grams of Blankit T (or 10 cc. of the above prepared hydrosulphite liquor); allow to stand for 15 minutes, then run in 100 cc. of the stock indigo solution by means of a long-tubed funnel. Stir gently and allow to stand for 30 minutes. When the liquor is clear and of an amber-yellow color it is ready for use. Take a test-skein of cotton yarn which has been boiled out and squeezed (but not dried) and pass it through this indigo vat without heating. Take care to manipulate the dyeing so as to disturb the liquor as little as possible, as much exposure to the air will cause undue oxidation and considerable indigo will be precipitated in the vat. When the skein has become thoroughly and evenly saturated with the liquor, squeeze it out well, and then expose the skein to the air for 5 or 10 minutes. When the skein first comes from the vat it should be of a yellowish green color; on exposure to the air it soon turns blue. The dyed skein is then washed well in water and afterwards in a warm soap solution in order to remove all alkali and unfixed dyestuff (420). Dye a second test-skein of cotton yarn in a similar manner, but after oxidizing in the air give it a second passage through the indigo vat and oxidize again, after which wash and soap. This will represent the color obtained by two dips (421). In the same manner dye a third skein, giving it four dips (422). Also dye skeins of woolen yarn in the same manner, giving one dip (423), two dips (424) and four dips (425). For the woolen yarn use a

first wash water acidulated with a little sulphuric acid in order to neutralize the alkali; then wash well again in plain water and finally soap. If the dye-vat turns bluish owing to oxidation a fresh quantity of hydrosulphite must be added, the liquor stirred gently and allowed to stand for 15 minutes. To maintain the proper dyeing strength of the vat fresh additions of the stock solution of indigo are made from time to time as needed. If too much hydrosulphite is present in the vat the color will not be well taken up and the blue will not develop quickly on exposure to the air.

The hydrosulphite vat for indigo is the simplest and the most popular method of applying this dyestuff at the present time. Other forms of vats, depending on the nature of the reducing agent, have been used. The *fermentation vat* is the oldest form of indigo dyeing and is still used to a considerable extent in wool dyeing. Its operation depends on the reducing action of certain ferments, and it is prepared with bran, woad, and madder. The woad is supposed to furnish the particular ferment, while the bran and madder serve as nourishment for the growth of the ferment. The alkali employed is lime, which serves the double purpose of neutralizing the acid liberated in the fermentation and providing the alkali necessary for the solution of the reduced indigo. The fermentation vat is difficult to prepare and also difficult to maintain in proper working condition. The *copperas vat* employs ferrous sulphate as the reducing agent and lime as the alkali. It is a cumbersome and unsatisfactory method and is not used at present. The *zinc vat* uses zinc dust for the reducing agent and either lime or caustic soda as the alkali. It is quite an efficient form of vat and is still employed considerably in cotton dyeing. All of these vats, however, contain a large amount of sediment, and care must be taken in dyeing not to disturb this sediment or it will get into the goods being dyed. There is also considerable loss of indigo in these vats, whereas in the hydrosulphite vat there is no sediment, and the loss of indigo is exceedingly small.

Test the fastness of the indigo dyeings to light, washing, fulling, acids, and chloring (on cotton).



**Experiment 134. Use of Thio-Indigo Red.** — Stir 50 grams of Thio-Indigo Red B paste with 200 cc. of water, add 10 cc. of caustic soda solution of 76° Tw., then gradually stir in 10 grams hydrosulphite powder (Blankit T). Heat to 120° F. and allow to stand for 2 hours, or until the reduction is complete and the solution is of a yellow color. Dilute to one litre and preserve as a stock solution for dyeing. For the dye-vat take 400 cc. of water of a temperature of about 100° F., add 20 grams of salt, a small quantity (0.1 gram) of hydrosulphite powder and a few drops of caustic soda solution (76° Tw.). Stir well, allow to stand for 15 minutes and then add 100 cc. of the stock dye-solution. Stir gently and allow to stand for one hour, when the vat should be of a clear yellow color and ready for dyeing. Steep a test-skein of cotton yarn in this vat for 15 minutes, then squeeze and oxidize in the air for 30 minutes (426). Dye another skein in the same manner, giving it three dips (427), and a third skein, giving it five dips (428). The addition of salt is for the purpose of causing a more rapid fixation and better exhaustion of the dyestuff. The bath may be strengthened by further additions of the stock solution. If the bath becomes red and loses its clear yellow color a little more hydrosulphite powder should be added, and the liquor stirred gently and allowed to stand for 15 to 30 minutes to allow it to become thoroughly reduced again.

Prepare a vat with Thio-Indigo Scarlet R in the same manner as above and make dyeings with one dip (429), three dips (430), and five dips (431).

When the dyeings have been exposed to the air sufficiently to become thoroughly oxidized, they should be well washed in water and then in warm soap solution to remove all unfixed dyestuff and chemicals.

Test the fastness of the two dyestuffs to light, washing, and bleaching.

**Experiment 135. Use of Indanthrene Blue.** — Prepare a dye-bath with 400 cc. of water, 8 cc. of caustic soda solution (53° Tw.), and 8 grams of Blankit T. Heat to 120° F., and then stir in 2 grams (20 per cent.) of Indanthrene Blue GCD. When

the liquor is clear and shows no undissolved particles (test by dropping on a piece of filter paper), the bath is ready for dyeing. Dye a test-skein of cotton yarn in this bath for one hour at 120° F. Keep the cotton beneath the liquor and expose the bath as little as possible to the action of the air. After dyeing rinse well in water, then in water slightly acidulated with sulphuric acid, and finally in a dilute soap bath (432). Test the fastness of this color to light, washing, and bleaching.

The indanthrene colors are best applied in mechanical dyeing apparatus so that the liquor during circulation comes into contact with the air as little as possible. The indanthrene dyestuffs are somewhat different in their behavior than the indigo dyes, as they exhaust very well from the bath and the color dyes up directly on the fibre and does not require a subsequent oxidation. On this account the amount of coloring-matter to be used may be based directly on a percentage of the material dyed.

**Experiment 136. Use of Indanthrene Yellow.**— Prepare a dye-bath as above, using 10 per cent. of Indanthrene Yellow G, and dye a test-skein of cotton yarn for one hour at a temperature of 140° F. It will be noticed that this dyestuff on reduction gives a blue solution and that the cotton is blue in color when first taken from the dye-bath. For the better development of this color, after dyeing, squeeze, rinse in water, and then pass through a very dilute cold solution of chrome (0.1 gram per litre). This facilitates the oxidation of the color very materially. Finally wash well and soap as usual (433). Test this color for fastness to light, washing, and bleaching.

**Experiment 137. Production of Fast Pink with Indanthrene Dyes.**— Prepare a dye-bath as in Exp. 135, using 3 per cent. of Indanthrene Red B, and dye a test-skein of cotton yarn for one hour at 140° F. Wash well and rinse in dilute acid, and finally soap (434). This color gives a rather bright pink when used in small percentages and the color is very fast to light, washing, acids, and bleaching. By combining with small amounts of Indanthrene Yellow R, bright yellowish pinks may be obtained.

**Experiment 138. Use of Ciba Blue.** — Prepare the dye-vat as follows: Make a paste with 0.5 gram of Ciba Blue 2B (powder), 1 cc. caustic soda (76° Tw.) solution, and some hot water; also dissolve 2 grams of hydrosulphite powder (Blankit T) in 15 cc. of cold water and 1 cc. of caustic soda solution. Then add this hydrosulphite solution to the dyestuff, dilute to 400 cc. with hot water and slowly boil. The dye-vat should then be completely reduced and be of a golden yellow color. Dye a test-skein of cotton yarn in this bath for 30 minutes at 170° F., squeeze well, and allow to oxidize in the air for 15 minutes; then rinse well in cold water, and finally work in a boiling dilute soap bath (435). The treatment with the boiling soap bath very materially brightens the color, and also gives it greater fastness to washing and bleaching. A still greater fastness to bleaching may be obtained by an after-treatment with bluestone in the usual manner.

#### NOTES.

1. **Classes of Vat Dyes.** — The vat dyes include *indigo*, *thio-indigo dyes*, *indanthrene dyes*, *ciba dyes*, and *algol dyes*. With the exception of indigo they are all of comparatively recent introduction. The dyestuffs themselves are insoluble in water but when reduced they yield products which are soluble in alkalies. Therefore the dye-bath consists of a mixture of the dyestuff, a strong reducing agent, and an alkali solution; such a mixture is termed a vat. Indigo is a blue dyestuff; the thio-indigo dyes include a purplish red and a scarlet; the indanthrene dyes comprise blue, violet, gray, yellow, brown, green, and dull red coloring-matters; among the ciba dyes are to be found blue, violet and red colors, as also among the algol dyes. The vat dyes are in general characterized by great fastness to light, washing, acids, alkalies, and in some cases to bleaching with chloride of lime. This makes them very valuable products, but at the present time, with the exception of indigo, they are very expensive. The vat dyes may be applied to all fibres, but they are probably more suited to the dyeing of cotton on account of the alkaline nature of the dye-vat. The material to be dyed is simply immersed in

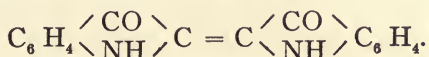


the dye-bath or vat until thoroughly impregnated with the solution; it is then squeezed and exposed to the air, which causes the oxidation of the reduced "leuco" compound and the formation of the color. The temperature of the vat is usually lukewarm for the purpose of facilitating the impregnation of the fibre with the solution. In some cases the dipping and oxidation have to be repeated several times in building up a heavy color.

**2. Indigo.** — This is one of the oldest dyestuffs of which we have any knowledge, and even at the present time it is perhaps the most important and extensively used of all dyes. Up to within rather recent years indigo was obtained from the indigo plant extensively cultivated in India and other Asiatic countries, as well as the tropical countries of Central and South America. Previous to the introduction of the oriental indigo (about 1500 A.D.) into Europe, the same dye had been extracted from a plant known as *woad* (*Isatis tinctoria*). The indigo plant is botanically known as *Indigofera*, and there are several species, the more important of which are *I. tinctoria*, *I. anil*, *I. disperma*, and *I. argentea*. Woad is still used to some extent in the preparation of certain vats, but only in connection with indigo, and never by itself. The coloring principle present in the indigo plant is known as *indigotin*, or indigo blue. The crude product, however, contains several other substances in varying amounts, such as indirubin or indigo-red, indigo-brown, indigo-glutin, and some mineral matters. The process of extracting indigo from the plant is rather complicated. The plant is cut when ripened, the leaves are stripped off and steeped for some time in vats of water. By a process of fermentation the coloring-matter which exists in the plant as a glucoside (*indican*) is changed to a soluble form and dissolves in the water, yielding an olive green solution. The liquor is run off into another vat, where it is beaten or churned up so as to expose it to the oxidizing action of the air. This causes the precipitation of the insoluble indigotin as a fine blue mud. This is collected, dried, and pressed into small bricks or cubes about three inches in size, in which form it appears in trade. At the present time indigo is also

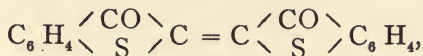
prepared in large quantities from coal-tar products by synthetic processes. There are several methods employed; the principal one uses naphthalene as the starting point. The synthetic indigo is rapidly replacing the natural product, as it can be made cheaper, purer, and more uniform. Chemically it is identically the same as the indigo derived from the plant. Indigo is extensively used for both wool and cotton dyeing, though it is being used proportionately less for the dyeing of wool since the introduction of the fast alizarin and anthracene blue dyes. It is not much employed for the dyeing of silk. In calico printing it has an extensive application, principally for discharge styles.

The chemical formula of indigo is rather complicated, but it has been definitely established as



In preparing the vat this indigotin is reduced to a leuco (or colorless) compound called indigo-white, which is readily soluble in alkalis (solutions of caustic soda, soda ash, lime, etc.). In this form it is readily absorbed by the fibre, in which it is oxidized again to indigotin (or indigo-blue) by the action of the air.

**3. Thio-Indigo Dyes.** — These are synthetic dyestuffs closely related to indigo in chemical composition and, as the name would indicate, containing sulphur. These dyes are of very recent discovery and comprise two commercial products, Thio-Indigo Red B and Thio-Indigo Scarlet R. The chemical formula for the red dye has been determined as



from which its relation to indigo may be readily seen. These dyes come into the market in the form of pastes containing 20 per cent. of coloring-matter, and are dyed in vats prepared about in the same manner as for indigo. Thio-Indigo Red is also soluble

in sodium sulphide with reduction, and hence may be used in practically the same manner as a sulphur dye, but the color so obtained is not as satisfactory as when produced from the vat.

**4. The Indanthrene Dyes.** — These dyes are not derivatives of indigo, but are prepared from amido-anthraquinone compounds. They include quite a range of colors at the present time and are being constantly added to, though a bright red and green have not yet been found. The list at the present time includes:

- Indanthrene Blue GCD.
- Indanthrene Blue RS, RC, RSP, RZ, and RRZ.
- Indanthrene Violet RT, R extra.
- Indanthrene Dark Blue B, BB, and BO.
- Indanthrene Maroon R.
- Indanthrene Gray B.
- Indanthrene Yellow G and R.
- Indanthrene Orange.
- Indanthrene Green.
- Indanthrene Copper.
- Indanthrene Brown B.
- Indanthrene Red B.
- Indanthrene Olive G.

These dyes are brought into solution in caustic soda by reduction with sodium hydrosulphite. In a vat thus prepared, however, they act very much after the manner of substantive dyes on cotton, some of them being dyed almost at the boil, the dyeing and development of the color taking place almost simultaneously, and the dye-bath becoming practically exhausted. Some of these dyes, however, must be applied at lower temperatures, and require subsequent oxidation in order to develop the color. The indanthrene dyes are brought into trade in the form of pastes containing 20 per cent. of coloring-matter. For the production of heavy shades large proportions of the pastes must be taken.

**5. The Ciba Dyes.** — These are halogen derivatives of indigo and are applied in the same general manner. The blue dyes of this series are the most important, and possess excellent fastness to light, washing, and bleaching with chloride of lime.



## SAMPLES.

- 420. Indigo on cotton; one dip.
- 421. Indigo on cotton; two dips.
- 422. Indigo on cotton; four dips.
- 423. Indigo on wool; one dip.
- 424. Indigo on wool; two dips.
- 425. Indigo on wool; four dips.
- 426. Thio-Indigo Red on cotton; one dip.
- 427. Thio-Indigo Red on cotton; three dips.
- 428. Thio-Indigo Red on cotton; five dips.
- 429. Thio-Indigo Scarlet R on cotton; one dip.
- 430. Thio-Indigo Scarlet R on cotton; three dips.
- 431. Thio-Indigo Scarlet R on cotton; five dips.
- 432. Indanthrene Blue GCD on cotton.
- 433. Indanthrene Yellow G on cotton.
- 434. Indanthrene Red B on cotton.
- 435. Ciba Blue on cotton.

## QUIZ 22.

667. What is meant by a "vat" dye? How does this class in general differ from other classes of dyestuffs?

668. Name the principal members belonging to the group of vat dyes. What range of color can be obtained with these dyes?

669. What is the general principle of applying the vat dyes? What fibres are mostly dyed with these colors?

670. What are the general characteristics of the vat dyes as to fastness? What is the chief drawback in the general use?

671. What is meant by a "leuco" compound? Why do the vat dyes in general require to be oxidized?

672. In dyeing with vat colors, how are heavy shades generally built up? Does the dye-bath with vat colors usually exhaust well?

673. What is "hydrosulphite"? Name some of the commercial preparations of this substance. What is its chemical function?

674. How is a solution of sodium hydrosulphite prepared? Give the chemical reaction which takes place. Is this solution stable?

675. Give some historical information concerning indigo. What was its source in former years, and what is its chief source at the present time?

676. How is indigo obtained from the plant? What is the coloring principle of indigo? What other substances exist in natural indigo?

677. What is the chemical formula of indigo? What is indican; indigo-white?

678. Give an outline of the general method of preparing the indigo vat with hydrosulphite.

679. Give the general method of dyeing cotton and wool in the indigo vat. What temperatures are employed, and how is the material treated after dyeing?

680. What other forms of vat are employed for the dyeing of indigo? Give the reducing agents and the alkalies used.

681. What can you say regarding the fastness of indigo blues to light, washing, acids, and bleaching?

682. What is Thio-Indigo Red? How is it applied in dyeing? What are its drawbacks as a dyestuff? What is its general fastness?

683. Name some of the indanthrene series of dyes. How are they applied in dyeing? What amounts of dyestuff are necessary to yield full shades?

684. How does the fastness of Indanthrene Blue compare with that of indigo?

685. How may a pink fast to washing and bleaching be obtained on cotton with indanthrene dyes?

686. What are the ciba dyes? Give the method of their application. How does Ciba Blue compare with Indanthrene Blue?

## SECTION XXIII.

### THE TESTING OF DYESTUFFS.

**Experiment 139. To Obtain the Money Value of a Dyestuff Sample.** — In the testing of a dyestuff sample for its money value it is, of course, necessary to test it in comparison with another sample of the same (or a strictly similar) dyestuff of a known or established money value. Take two samples of Wool Blue and prepare solutions of the same containing 2.5 grams per litre, labelling them "A" and "B." Prepare two dye-baths containing the same amount of water, acid, and Glaubersalt, and add 10 cc. of the respective dyestuff solutions. Then dye two test-skeins of woolen yarn identical in character and weight in these baths, maintaining carefully the same conditions as to temperature and time of dyeing in both cases. After dyeing in the usual manner for 45 minutes, remove the two skeins, squeeze the excess of liquor back into the respective dye-baths, and then dry up portions of the two skeins. Now compare these for depth of color and set aside the heavier shade for comparison; continue dyeing the weaker shade, adding to the dye-bath sufficient of its respective color to bring the shade to a match with the other sample. The amounts of the two dyestuff solutions used to produce the same depth of shade will be inversely proportional to the values of the respective dyes, so if the actual money value of one of the samples is known it is a simple matter to calculate the relative value of the second. For example, suppose that on the first dyeing, sample "A" proved to be strongest dyestuff; on continuing the dyeing of "B," it was necessary to use 2.5 cc. more dye solution to match "A"; further, suppose that sample "A" was priced at 42 cents per pound. What would be the relative money value of "B"?

Dye solution used for "A,"	10 cc.
Dye solution used for "B,"	12.5 cc.



Then  $12.5 : 10 = 42 : x$

and 
$$x = \frac{10 \times 42}{12.5} = 33.6 \text{ cents per pound.}$$

This method of testing the comparative strength or value of dyestuffs may be carried to a rather high degree of accuracy, but it is necessary that the comparative dye-tests be made under exactly the same conditions in every respect, and also that the eye be trained to match the depth of colors with great accuracy. The amounts of the dye solutions should be accurately measured by means of a graduated pipette, and exactly the same amounts of sulphuric acid and glauber salt should also be taken by means of definitely measured solutions; and further the total volume of each dye-bath should be the same. To insure the most accurate results, it is best to carry out a second dyeing of the weaker sample, using the required amount of its solution added to the bath all at one time. This is especially desirable if the second sample has been brought to a match by a number of successive additions of the color solution. For instance, in the example quoted above, suppose the several additions of the color solution of "B" to have been as follows:

First, 10 cc.	Fourth, 0.5 cc.
Second, 1 cc.	Fifth, 0.3 cc.
Third, 0.5 cc.	Sixth, 0.2 cc.

making in all 12.5 cc. It would be better to then dye another skein, using 12.5 cc. as a first addition to the dye-bath. It will frequently be found that a slightly increased amount of the dye solution will be required to bring this second test to a match. This is accounted for by the fact that the prolonged dyeing necessitated by the numerous additions to the bath will cause an abnormal absorption of dyestuff.

If properly carried out, this method of analysis is capable of giving results accurate to within at least two per cent., provided the samples being tested are the same kind of dyestuff. If, however, the dyes are not quite of the same tone of color, some difficulty may be experienced in judging accurately the point

at which the samples are matched and considerable skill in matching will be required to arrive at their proper valuation. The matching in this case may be usually rendered somewhat easier by observing the colors through red, blue or yellow glasses, which have the effect of cutting out certain undesirable tones. Furthermore, the eye is more sensitive to differences in intensity of some colors than in others; for instance, it is quite difficult to detect small differences in the depth of yellow colors, whereas in blues or reds, small differences are easily detected. Violet colors and reddish tones of blue are also rather difficult to accurately match; and dull and broken tones of any color are harder to approximate than clear and bright tones. It is not well to employ too heavy shades for comparison, or the accuracy of the method will be much impaired.

As sometimes one dyestuff may exhaust better in the first bath than another corresponding dye, or even with the same dyestuff, it is at times possible to mix with it some chemical to cause it to exhaust better than when pure, in the practical testing of dyes it is best to make an *exhaust test*. This is done by diluting the dye-bath used for the first dyeing to its original volume, and without the further addition of dyestuff or reagents, to dye a second test-skein. The intensity of the dyeing thus obtained will measure the degree of exhaustion of the dyestuff in the first bath.

It is not well to employ too heavy shades for comparison; as a rule, from one-half to one per cent. dyeings will be found quite satisfactory for most colors. In the case of black dyes, however, where it is necessary to obtain a comparison of the tone of black produced, it will, of course, be necessary to use more than one per cent. of dyestuff.

In cases where the test-skeins must be mordanted or otherwise treated before or after the dyeing, care must be taken that the several skeins employed in the tests receive exactly the same manner and degree of treatment. In order to insure the proper conditions it is best to mordant all the test-skeins used simultaneously and together in the same bath; and

this should also be done where any after-treatment of the dyeing is required.

Make a comparative test of two samples of a substantive cotton color; two samples of a basic color for cotton, mordanting with tannin-antimony; two samples of an alizarin color for wool on a chrome mordant; and two samples of an anthracene color for wool, after-mordanting with chrome. In all these cases make exhaust tests on the same dye-bath.

**Experiment 140. To Determine if a Dyestuff is Simple or Mixed.** — A large number of the dyestuffs on the market are not simple or single coloring-matters, but consist of two or more dyestuffs mixed together. This mixing of colors is practised for the purpose of altering the tone of the dyestuff; or for the forming of various colors, such as the production of a green by mixing a blue and a yellow dye. It is also practised for the purpose of adulterating various dyes with others of cheaper quality.

The presence of mixtures in a dyestuff, however, must not always be taken as evidence of sophistication. In the manufacture of dyestuffs it often happens that successive lots of the same coloring-matter do not exhibit precisely the same tone, but it is very desirable to the dyer that the dyestuff as sold should always be of exactly the same tone. The manufacturer, therefore, adopts a standard, and tones the various lots to match this standard by the proper addition of suitable but similar dyes. Therefore a dyestuff may be a perfectly true article and yet show evidence of mixed colors. The amount of mixture, however, under these circumstances is very small; whereas admixture for purposes of sophistication is usually rather large.

A simple test of considerable practical value for detecting a mixture of dyes is as follows: Moisten a small sheet of paper with water, place a little of the dyestuff on one end of the paper and then blow the breath across it so as to scatter the dyestuff in fine particles over the paper. A mixture of dyes will generally give a mottled appearance of several colors. A modification of this test, which at times will yield better results, is to place some



concentrated sulphuric acid in a porcelain dish, and then sprinkle a little of the suspected dyestuff over the surface of the acid. If particles of different dyes (even though they may be of the same color originally) are present they will generally show different colors on contact with the acid, causing them to be easily detected. Prepare a green dyestuff by making an intimate mixture of one part Naphthol Yellow and three parts Wool Blue, and test the mixture so obtained by the two methods just given.

A further method of testing depends on the difference in the capillarities of the two dyestuffs when in solution. A portion of the suspected dyestuff is dissolved in water and a single drop of the solution placed on a piece of filter (or blotting) paper. If the dyestuff is a mixture, two rings of color will generally be observed as the drop of solution spreads out over the paper. Test the green mixed dyestuff, as prepared above, in this manner, and observe the result. Sometimes this test may be rendered more distinct by using an alcoholic solution of the dyestuff. Make this test with a drop from an alcoholic solution of a mixture of Methyl Violet and Safranine.

**Experiment 141. To Determine the Class to which a Dyestuff Belongs.** — It is often desirable to ascertain the chemical character of a sample of unknown dyestuff; that is to say, the classification of a coloring-matter with reference to its dyeing properties. With reference to these properties, nearly all dyes may be classified into four general groups, as follows:

(a) Acid dyes, including those that are dyed in an acid bath, and which consist of the salts of color acids.

(b) Basic dyes, including those that are dyed in neutral or alkaline baths, and which consist of the salts of color bases.

(c) Substantive dyes, including those that dye both animal and vegetable fibres, and which consist principally of benzidine and allied derivatives.

(d) Mordant dyes, including those that do not dye either the animal or vegetable fibres directly, but which form color-lakes with various metallic oxides. These dyes consist mostly of anthracene derivatives and allied compounds of an alcoholic

nature. This classification, however, must not be taken as absolute and rigid, as one class may merge into another in almost an imperceptible manner, and there are dyestuffs which exhibit the characteristics of more than one; for instance, there are dyes which may be dyed in an acid bath, and would consequently be considered as acid dyes, but which also dye on metallic mordants, and hence would also be included among the mordant colors.

Again, basic dyes may also be dyed in baths more or less strongly acid; and substantive dyes may be dyed (on wool, for instance) from neutral, acid, or alkaline baths, or may even be dyed on mordants. So it may be seen that it is not such a simple matter, after all, to quickly decide as to what class a dyestuff is to be referred. This problem can only be solved by a series of systematic tests, which should be carried out in the following manner:

A solution of the dyestuff should be made of a strength of about 5 grams per litre, using distilled water as the solvent. The solution is best made by first boiling the coloring-matter with about 200 cc. of the water for ten to fifteen minutes, and then diluting the solution to one litre by the addition of cold water. The following dye-tests are then carried out with this solution:

(a) A test-skein of scoured woolen yarn is mordanted by boiling for one-half hour in a bath containing 3 per cent. of chrome and 4 per cent. of tartar; washed well, and then dyed in a bath containing 1 per cent. of the dyestuff. If a skein of woolen yarn becomes dyed, and especially if most of the color is extracted from the bath, the dyestuff in question may belong to the mordant-dyeing class, though the certainty of this can only be ascertained by the succeeding tests. If, however, the skein in this test does not become dyed, which is hardly likely, then it is positively known that the coloring-matter under examination is not a mordant dye.

(b) A second test-skein of woolen yarn is dyed in a bath containing 10 per cent. of Glaubersalt and 4 per cent. of sulphuric

acid, together with 1 per cent. of the dyestuff; the material is boiled for one-half hour, then squeezed out and washed well. If the wool remains undyed in this case, but was dyed in test (a), almost positive assurance is afforded that the coloring-matter in question is a mordant dyestuff. If, however, the wool is dyed, the coloring-matter may be either an acid dyestuff belonging to the after-chromed variety, or it might also be a basic or a substantive dye. This must be determined by the subsequent tests.

If the result of these two tests leads to the indication of a mordant dyestuff, this may be confirmed by boiling a few cubic centimeters of the dye solution with separate solutions of chromium acetate and aluminium sulphate; if the dyestuff belongs to the mordant class, there will be a precipitate of a color-lake in each case. If there is no precipitate, this would indicate that any dyeing obtained on the mordanted wool in test (a) does not proceed from the presence of a mordant dye. If the skeins are dyed in both tests (a) and (b), and the dyestuff solution also causes the precipitation of a color-lake with the salts of chromium and aluminium, then it may reasonably be concluded that the coloring-matter in question is a mordant dyestuff which also dyes wool from an acid bath. If no color-lake, however, is formed with the metallic salts, then the dyestuff is probably an acid color.

(c) A test-skein of woolen yarn is dyed in a bath containing 1 per cent. of the dyestuff and 10 per cent. of Glaubersalt, being boiled for one-half hour, then squeezed and washed well. If the wool remains undyed in this case, it indicates a mordant dye, as in the previous test (there are, however, certain substantive dyes which leave wool practically undyed under such conditions, and consequently, before judging definitely that the dyestuff belongs to the mordant class, the fact should be confirmed, as before described, by the precipitation of the metallic color-lake). If the skein remains undyed in this test, but was dyed in test (b), the dyestuff is probably an after-treated mordant dye. If the skein is dyed in test (c), it may be either an acid, substantive, or basic dye. If the first, it would also have been dyed in test (b).

(d) A skein of cotton yarn is dyed in a bath containing 1 per



cent. of the dyestuff and 10 per cent. of common-salt; boil for one-half hour, then squeeze, and wash well in water, and then in a dilute lukewarm soap bath. If the cotton is not dyed, the dye may belong to either the mordant, the acid, or the basic class. If to the first, its nature would have already been indicated by the previous test. If to the second, it would also have dyed the wool in test (b), and probably in test (c). If to the third, the wool in test (c), and probably also in test (b), would have been dyed. If the cotton skein, however, in this test is well dyed and retains its color after soaping, it indicates that the dyestuff under examination belongs to the substantive class.

(e) A skein of cotton yarn is worked in a bath containing 4 per cent. of tannic acid for one-half hour at 180° F., then squeezed and worked for ten minutes in a cold bath containing 2 per cent. of tartar emetic, then squeezed and well washed. This mordanted cotton skein is then dyed in a bath containing 1 per cent. of the dyestuff for one-half hour at 160° F., after which it is squeezed and well washed. If the cotton becomes dyed in this test, and the dye-bath is rapidly and rather completely exhausted, the dyestuff may be regarded as a basic dye, in which case the wool in test (c) would also be dyed, though with some basic dyes only slightly so, and the cotton in test (d) would remain practically undyed. If the cotton skein in this test remains undyed or is only slightly dyed, the dye may be either an acid or a mordant color, the distinction between which would have already been made in the previous tests.

**Experiment 142. Chemical Method of Distinguishing between Acid and Basic Dyestuffs.** — These two classes of dyes may be rather easily distinguished by certain chemical tests as follows:

(1) Basic dyes are not removed from an acidulated aqueous solution by agitation with ether, whereas acid dyes are taken up by the latter. Carry out the test as follows: Take a small quantity of Acid Violet (a portion the size of a grain of wheat is sufficient), and dissolve in about 5 cc. of dilute sulphuric acid; then add 5 cc. of ether and shake well. After settling, notice that the layer of ether has taken up the coloring-matter, showing the

presence of an acid dye. Repeat the test, using Methylene Blue, and notice that the ethereal layer is not colored. A sample containing a mixture of acid and basic dye (which, however, is hardly likely, as dyes of different classes are seldom mixed together) may be completely separated by this test, the acid dye being completely extracted by the ether.

(2) Caustic soda precipitates most basic dyes from their aqueous solutions (the safranine class excepted), whereas acid dyes are not so precipitated. Take about 5 cc. of an aqueous solution of Acid Violet in a test-tube, add about 5 cc. of caustic soda solution (1:10) and warm gently. Notice that the solution remains clear. Repeat the test, using a solution of Magenta, and notice that a precipitate is formed. Repeat the test again, using a dilute solution of Safranine, and notice that no precipitate is produced. (In the latter test, if a concentrated solution of Safranine is used, a precipitate will form.)

(3) Aqueous solutions of basic dyes are precipitated by addition of the so-called "tannin reagent," whereas acid dyes are not so precipitated. This is probably the best means of separating basic from acid dyes. The tannin reagent is prepared by dissolving 25 grams of tannic acid and 25 grams of sodium acetate in 250 cc. water. Add a few drops of this reagent to a dilute (1 per cent.) solution of Acid Violet and warm gently; notice that no precipitate is formed. Repeat the test, using a dilute solution of Magenta; the latter will produce a precipitate.

Test several samples of unknown dyes in order to determine whether they belong to the acid or the basic class.

#### **Experiment 143. Detection of Adulterations in Dyestuffs. —**

Commercial dyestuffs are frequently adulterated with common-salt (sodium chloride), glauber salt (sodium sulphate), soda ash (sodium carbonate), dextrin, starch, sugar, and Epsom salts. The presence of such substances, however, does not always indicate intentional adulteration; for in their manufacture many dyes are "salted out" from solution, or precipitated by strong brine solutions, and therefore would nearly always show the presence of varying amounts of common-salt. Again, in the

manufacture of dyestuffs, it is desirable to prepare products of uniform strength, and as the different lots of manufactured dye seldom show exactly the same strength, a definite standard must be adopted to which weaker lots are brought up by the addition of a stronger dye while too strong a lot of dyestuff is diluted to the standard by the addition of suitable neutral salts. The occurrence of a large proportion of the above-mentioned salts in the dyestuff must, however, be taken as indicating intentional adulteration. For basic dyes sodium chloride and dextrin are chiefly used; for the acid dyes sodium sulphate is employed, and for the substantive dyes either sodium chloride or sulphate may be used. Dextrin in some cases is added to increase the solubility of the dyestuff.

(A) *Detection of Sodium Chloride.*—As silver nitrate forms an insoluble white precipitate of silver chloride when added to a solution of common-salt, this reagent is used for the test. In many cases the test may be carried out by simply dissolving a small quantity of the dyestuff in water, adding a few drops of nitric acid (to prevent the precipitation of any other salt of silver besides the chloride), and then a few drops of a solution of silver nitrate, when the production of a white precipitate will indicate the presence of a chloride. This method, however, is not always reliable, as many dyestuffs are hydrochlorides of color-bases (basic dyes), or give insoluble salts of silver, in which cases the formation of a white precipitate would not necessarily indicate the presence of common-salt. It is best to ignite a portion of the dyestuff in a porcelain crucible, so as to burn off all volatile and organic matter, leaving only mineral matter in the ash. Dissolve the ash in water, add a few drops of nitric acid and then the silver nitrate; a white precipitate of silver chloride will be formed if common-salt is present. A few dyes, such as the eosins, leave chlorides (or bromides or iodides) in the ash after ignition; hence this test would not be satisfactory. For such dyes, the aqueous solution of the coloring-matter should be acidulated with dilute sulphuric acid, then shaken up with ether. The dyestuff will be dissolved out by the



ether, leaving any common-salt which may be present in the aqueous layer. The latter may be removed and tested with silver nitrate as described above. If the dyestuff is soluble in alcohol, the coloring-matter may first be extracted by warming with this solvent, and the test for common-salt may then be made with the residue.

(1) *Testing for Salt in Acid Yellow.* — Dissolve a small portion of the pure dyestuff in water, add a couple of drops of dilute nitric acid and a few drops of silver nitric solution; no precipitate will be produced. Repeat the test, using a sample of the dyestuff containing common-salt, and notice the formation of a white precipitate of silver chloride (which will be more or less colored by the dyestuff).

(2) *Testing for Salt in Magenta.* — Dissolve a small quantity of the pure dye in water, and test with silver nitrate as above. As the dye itself is the hydrochloride of the color-base, a precipitate of silver chloride will be formed, though no common-salt is present. Place a small quantity of the dyestuff in a porcelain crucible, and ignite until all organic matter is completely burned away; dissolve the ash in a small quantity of water acidulated with a few drops of nitric acid, and test with silver nitrate; no precipitate will be produced. Repeat this test, using a sample of Magenta containing common-salt and note the formation of a precipitate of silver chloride.

(3) *Testing for Salt in Eosin.* — Place a small quantity of pure Eosin in a porcelain crucible and ignite as above described; on dissolving the ash in water and testing with silver nitrate in the usual manner, a precipitate will be formed though no common-salt is present. Next take a small quantity of the Eosin, dissolve in 10 cc. of water in a test-tube (or better, a stoppered separatory funnel), add 5 cc. of ether (be sure the solution is *cold* before adding the ether), shake well and allow to stand. The ethereal layer, containing the dyestuff in solution will collect on top, while the aqueous layer will remain at the bottom. If the color is not well extracted from the bottom layer pour off the ethereal layer and repeat the extraction with

fresh ether. Withdraw the aqueous layer by means of a pipette, and test it with silver nitrate as usual; no precipitate will be produced. Repeat this test, using a sample of Eosin containing common-salt, and notice that a precipitate of silver chloride will be formed.

(4) *Testing for Salt in Orange II.* — Place a small sample of the pure dye in a test-tube and dissolve in 10 cc. of warm alcohol; it should be completely soluble. Repeat this test, using a sample of the dye containing common-salt; notice that a residue is left. Dissolve this in water and test with silver nitrate as usual; a precipitate of silver chloride will be produced.

(B) *Detection of Sodium Sulphate.* — Sulphates in general are detected by the addition of barium chloride to their solution, whereby an insoluble white precipitate of barium sulphate is formed. The presence of sulphates in the ash of a dyestuff does not necessarily indicate adulteration with Glaubersalt, as many dyes are themselves sulphonated compounds, and on ignition leave sodium sulphate in the ash. The best procedure for testing for Glaubersalt in a dyestuff is as follows: Dissolve some pure Benzopurpurin in a small amount of water, add a few drops of dilute hydrochloric acid (to prevent other compounds of barium being precipitated), then a solution of barium chloride as long as a precipitate forms. This precipitate, which consists of barium sulphonate, is filtered off, washed, and boiled with a solution of ammonium carbonate. This converts it into barium carbonate; filter again, and wash the residue of barium carbonate, and then add dilute hydrochloric acid to the latter, when it should be completely dissolved. Repeat the test, using a sample of Benzopurpurin containing Glaubersalt. The precipitate obtained with barium chloride, in this case, consists of a mixture of barium sulphonate and barium sulphate. On boiling this with ammonium carbonate, only the sulphonate is converted into barium carbonate, and on finally dissolving in hydrochloric acid, the barium sulphate will be left as an insoluble residue, thus showing the presence of Glaubersalt in the original dye. Another method for testing for sulphates is to precipitate the

dyestuff from its aqueous solution by the addition of pure common-salt to complete saturation. The precipitated dyestuff with excess of salt is filtered off, the filtrate acidulated with a few drops of hydrochloric acid and tested with barium chloride solution. This formation of a white precipitate will indicate the presence of glauber salt in the original dyestuff. This method, however, is not very satisfactory, as it is usually difficult to precipitate the dyestuff completely from its solution. Another method of testing for sulphates is to dissolve the dyestuff in strong warm alcohol (where this is possible), and as glauber salt is insoluble in alcohol it will be left as a white residue (as in the case of common-salt). This is to be dissolved in water and tested with barium chloride in the usual manner.

(C) *Detection of Soda Ash.* — This substance is frequently added to eosins, and sometimes to substantive dyes. It is detected by dissolving a sample of the dye in water, and adding hydrochloric acid to the solution, when an effervescence will be produced due to the liberation of carbon dioxide gas from the carbonate present. For example: Dissolve some pure Eosin in a little water and add a few drops of dilute hydrochloric acid; no effervescence will occur. Repeat the test, using a sample of Eosin containing sodium carbonate; a vigorous effervescence will be noted.

(D) *Detection of Epsom Salts.* — This consists of magnesium sulphate, and is occasionally added as an adulterant to dyes. The presence of the sulphate is detected in the manner described under the test for sodium sulphate. The presence of the magnesium is shown in the following manner: Place a portion of Methyl Violet containing magnesium sulphate in a porcelain crucible; ignite until all carbon and volatile matter is burned away. Dissolve the ash in hot dilute hydrochloric acid, and filter, if necessary. Neutralize the filtrate with ammonia water and add a solution of sodium phosphate. After standing for a short time a crystalline precipitate (of magnesium ammonium phosphate) is formed, showing the presence of a magnesium salt in the original dyestuff.



(E) *Detection of Dextrin.* — This impurity can usually be recognized by the peculiar odor dextrin gives on dissolving the dye in warm water. It may best be tested for as follows: Take a small sample of Methyl Violet containing dextrin; extract the coloring-matter with absolute alcohol; the dextrin will be left as a residue. Dissolve the latter in a small quantity of warm water, and notice the peculiar odor of the dissolving dextrin. Dextrin is frequently added to paste dyes and to basic dyes.

(F) *Detection of Starch.* — This impurity may be separated from the dyestuff by treating with cold water, in which the starch is insoluble. It may then be recognized as follows: Take a sample of Eosin containing starch; extract the coloring-matter with cold water; dissolve the residue of starch in a little boiling water, and add a few drops of a solution of iodine in potassium iodide. A deep blue color will be produced, indicating the presence of starch.

(G) *Detection of Sugar.* — This impurity is frequently added to crystalline dyes, as it occurs in the crystalline form itself. Its presence may be shown as follows: Take a sample of Magenta containing sugar; extract with absolute alcohol. The dyestuff passes into solution, whereas the sugar remains practically insoluble, and becomes nearly colorless. Heat the residue in a test-tube and notice the odor of caramel, indicating the presence of sugar.

**Experiment 144. Determination of the Capillary Speed of Dyestuffs.** — By the capillary speed of a dyestuff is meant the height to which its solution will rise in a given time through porous paper (filter or blotting paper). This factor is only a relative number, and is usually compared with pure water as a standard.

Take five strips of blotting paper measuring 5 inches in length and one-half inch in width; make a mark on each strip 1 inch from the end. Immerse one of these strips in a small beaker containing pure water so that the surface of the water comes exactly to the 1-inch mark on the paper. Sustain the strip in a perpendicular position, and allow it to remain in the water for

just one minute. Then measure the height to which the water has risen on the paper. Repeat the test with a fresh strip of paper, using a one per cent. solution of magenta, and after one minute measure the height to which the *color* has ascended. Repeat the test further on one per cent. solutions of Naphthol Yellow, Acid Violet, and Malachite Green. Tabulate the results obtained as follows:

Solution.	Distance color rises.	Compared with water as = 100.
Water.....	.....	100
Magenta.....	.....	.....
Naphthol Yellow S.....	.....	.....
Acid Violet.....	.....	.....
Malachite Green.....	.....	.....

### QUIZ 23.

687. Describe the method of determining the comparative money value of a dyestuff.

688. Suppose dyestuff "A" required 16 cc., "B" 10 cc., and "C" 12 cc. of their respective solutions to give the same depth of shade on testing; what would be the values of "B" and "C" if "A" is quoted at 36 cents per pound?

689. If several successive additions to the dye-bath have been made to secure a match, why is it best to repeat the dyeing using the required amount of dyestuff in a single addition?

690. What is meant by the "exhaust test" in valuing dyes, and what is its purpose?

691. What is the purpose of mixing different dyes together before sale? Does evidence of admixture always indicate adulteration?

692. Describe the tests given for the detection of mixed dyes.

693. What tests would you make to identify a basic dye; an acid dye; a substantive dye; a mordant?

694. What are the principal substances used for the adulteration of dyestuffs? Does the presence of common-salt in a dyestuff always indicate intentional adulteration? Why?

695. What is the general test for the detection of sodium chloride? Is this applicable to all dyestuffs?

696. How would you test for the presence of common-salt in Acid Yellow and similar dyestuffs?

697. How would you test for the presence of common-salt in Magenta and such dyes that consist of hydrochlorides?

698. Give the method for the testing of common-salt in the eosin group of dyes.

699. In case a dyestuff is soluble in alcohol, how may the presence of common-salt be shown?

700. What is the general method for the detection of sulphates? What other sulphate besides glaubersalt may be present in dyes?

701. Why should not the direct test for sulphates be made with dyestuffs? What is meant by a sulphonate?

702. Describe the method of testing for glaubersalt in a dyestuff.

703. How may glaubersalt be detected in a dyestuff by the "salting-out" process? In case the dyestuff is soluble in alcohol what process may be used?

704. How may the presence of soda ash be detected in a dyestuff? What classes of dyes are subject to this adulteration?

705. What is Epsom salts? How may its presence be shown in a dyestuff?

706. Dextrin is used as an adulterant with what classes of dyes? How may its presence be detected?

707. How is the presence of starch in a dyestuff detected?

708. What classes of dyes are adulterated with sugar? How is this substance detected?



## SECTION XXIV.

### CHEMICAL REACTIONS OF DYESTUFFS.

IN order to identify any particular dyestuff it is necessary to test it with various chemical reagents, whereby a series of chemical reactions are obtained, usually based on color changes. Tabulated reactions have been prepared of the various dyestuffs, and by reference to these it is usually possible to identify any individual coloring matter. Difficulty, however, will be experienced with coloring matters containing mixtures of two or more dyestuffs, as the reaction of one of the dyes in the mixture may obscure the reaction of the other dye. In the case of many mixtures it is practically impossible to determine accurately the exact dyestuffs present unless some method is available for the separation of the dyes.

Considerable evidence as to the identity of a dyestuff is furnished by its dyeing properties toward cotton and wool. In this manner it will be possible to determine if the dyestuff in question is acid or basic, etc., and this method of classification will eliminate many uncertain possibilities.

In the following experiments, to illustrate the results of the reactions given, the dyestuff Auramine is taken as an example.

**Experiment 145. Solubility Tests.** — (a) *With Water.* — Take about 0.1 gram of the dyestuff and add to 20 cc. of distilled water in a test-tube; shake well, and then boil; observe the relative solubility. Auramine is **very soluble**. (b) *With Alcohol.* — Repeat the test, using alcohol in place of water; note the relative solubility and the color of the solution. Auramine is **very soluble** with a yellow color. (c) *With Ether.* — Repeat the test, using ether; Auramine is **insoluble**. (d) *With Benzene.* — Repeat the test, using benzene; Auramine is **insoluble**.

**Experiment 146. Reaction with Sulphuric Acid.** — (a) *With Concentrated Acid.* — A small quantity (on the tip of a penknife

blade) of the dyestuff is added to about 5 cc. of pure concentrated sulphuric acid in a test-tube; shake well and note the color of the solution obtained. Auramine dissolves with **effervescence** (due to evolution of hydrochloric acid gas), and gives a **colorless solution**. (b) *On Dilution with Water*. — Add a drop or two of the strong acid solution as obtained above to about 10 cc. of water in a test-tube, and note the reaction which occurs. Auramine on dilution gives a **yellow color**. (c) *On Heating*. — The remainder of the strong acid solution is gradually heated to the boiling point. Auramine furnishes a **pale brownish yellow** solution.

**Experiment 147. Reaction with Hydrochloric Acid.** — Use an aqueous solution of the dyestuff containing 1 gram of dye per litre. To 5 cc. of this solution add 5 cc. of a solution of hydrochloric acid (containing 100 cc. of the strong acid per litre); allow to stand for 10 minutes, and note what reaction, if any, occurs. Auramine remains **unchanged**. Now heat the solution to boiling and note any change that occurs; Auramine becomes **decolorized**.

**Experiment 148. Reaction with Nitric Acid.** — To 5 cc. of the aqueous solution of the dyestuff add 5 cc. of a solution of nitric acid (containing 50 cc. of the strong acid per litre), and carry out the test as above. Auramine remains **unchanged** cold; on boiling it gives a **pale yellow solution**.

**Experiment 149. Reaction with Sodium Hydrate.** — Carry out the test as above described, using 5 cc. of a solution of sodium hydrate (containing 100 cc. of sodium hydrate of sp. gr. 1.3 to one litre). If a precipitate is formed, add about 2 cc. of ether, and shake; observe if the ether extracts the precipitated color from the caustic soda; then add a drop or two of acetic acid to the ethereal layer, and note any change. Auramine gives a **white precipitate**, extracted with ether, **becoming yellowish** on addition of acetic acid.

**Experiment 150. Reaction with Ammonia.** — Repeat the test as above given, using 5 cc. of commercial ammonia water. Auramine undergoes the same reactions as with sodium hydrate.

**Experiment 151. Reaction with Sodium Carbonate.** — Repeat the test as given above, using 5 cc. of a solution of sodium car-

bonate (100 grams per litre). If a precipitate is formed with the cold solution, heat to boiling and observe if this causes the precipitate to redissolve. Auramine gives a **milky yellow precipitate** which remains **insoluble on heating**.

**Experiment 152. Reaction with Tannin Reagent.** — Carry out the test, using 5 cc. of a solution containing 100 grams of tannic acid dissolved in 500 cc. of water and mixed with a solution of 100 grams of sodium acetate in 500 cc. of water. If a precipitate is formed heat to boiling and note any change in its character. Auramine gives a **yellow precipitate**, which on boiling becomes **brown, resinous, and partially soluble**. This reagent is useful for distinguishing between acid and basic dyes, as the latter alone give a precipitate.

**Experiment 153. Reaction with Alum.** — Add to the aqueous solution of the dyestuff 5 cc. of a solution of alum (containing 50 grams per litre). If a precipitate is formed, heat to boiling, and note any change that may occur. Auramine remains **unchanged** with the alum solution. Many of the acid dyes and nearly all of the mordant dyes give characteristic precipitates with alum.

**Experiment 154. Reaction with Potassium Bichromate.** — Add to the aqueous solution of the dyestuff 5 cc. of a solution containing 50 grams of potassium bichromate per litre. If a precipitate is formed, heat to boiling, and note any change that may occur. Auramine give a **yellow precipitate**, which on heating **becomes resinous** and for the most part **dissolves**. The majority of the basic dyes are precipitated by this reagent, as is also the case with most of the mordant dyes; only a few of the acid dyes are thus precipitated.

**Experiment 155. Reaction with Ferric Chloride.** — Add to the aqueous solution of the dyestuff 5 cc. of a solution containing 100 grams of ferric chloride per litre; note the reaction and then heat to boiling and observe any further change. Auramine remains **unchanged** in the cold solution; on heating the solution becomes **turbid** and of a **brownish yellow color**.

**Experiment 156. Reaction with Stannous Chloride.** — Add to the aqueous solution of the dyestuff 5 cc. of a solution containing



100 grams of stannous chloride per litre (with sufficient hydrochloric acid to yield a clear solution). After standing 10 minutes, heat to boiling, and note the reactions which occur. Auramine remains **unchanged**. A large number of dyestuffs either give characteristic precipitates, or become decolorized.

**Experiment 157. Reaction with Bleaching Powder.** — Add to the aqueous solution of the dyestuff 5 cc. of a solution of bleaching powder of 1.5° Tw. strength. After standing for 10 minutes, heat to boiling, and note any changes which occur. Auramine gives a **dirty, pale yellow precipitate**, which on heating turns to a **dirty red color**.

**Experiment 158. Reaction with Zinc Dust.** — Add to the aqueous solution of the dyestuff about 1 gram of zinc dust and 5 cc. of ammonia water; shake well and then heat to boiling; filter at once, and note if the filtrate if decolorized becomes colored again on exposure to the air. Auramine gives a **colorless** filtrate, and the color does **not reappear** on exposure to the air; but the precipitate left on the filter gradually becomes yellowish on standing. The solutions of nearly all dyes are decolorized by this reagent, and many dyes give a characteristic reappearance of color on exposure to the air. The loss of color is caused by the reduction of the coloring-matter, which may be converted either into a leuco (colorless) derivative (which allows of the original dyestuff being again formed on oxidation in the air), or be entirely destroyed. In the latter case no reappearance of color will be shown on oxidation.

**Experiment 159. Reaction with Zinc Dust and Acetic Acid.** — Repeat the above test as given, using, however, 5 cc. of acetic acid in place of the ammonia. Auramine becomes **blue** on acid reduction. Many dyestuffs give characteristic colors with this test, while some are decolorized completely. In many cases the original colors reappear on exposure.

Test the following dyestuffs in the same manner as outlined for Auramine:

Magenta	Benzopurpurin	Naphthol Yellow S
Acid Violet 4R	Alkali Blue	Alizarin Brown SO

TABULATION OF RESULTS WITH CHEMICAL REAGENTS.

Test.	1. Auramine.	2. Acid Violet 4R.	3. Alkali Blue R.
Solubility in { Water. Alcohol. Ether. Benzene.	Very sol. Very sol. Insoluble. Insoluble.	Quite sol. Quite sol. Insoluble. Insoluble.	Quite sol. Quite sol. Slightly soluble. Insoluble.
Concentrated sulphuric acid. { Solution. Dilution. Heating.	Colorless. Yellow. Brown-yellow.	Dark brown. Violet. Dark yellow-brown.	Red brown. Blue ppt. Dark brown.
Dilute hydrochloric acid. { Cold. Heating.	No change. Decolorized.	No change. No change.	Blue ppt. Blue ppt.
Dilute nitric acid. { Cold. Heating.	No change. Pale yellow.	No change. No change.	Blue ppt. Turns green.
Sodium hydrate. { Cold. Extraction with ether.	White ppt. Color extracted.	Colorless.	No change.
Ammonia. { Cold.	As above.	Colorless.	
Sodium carbonate. { Cold. Heating.	Yellow milky ppt. No change.		
Tannin reagent. { Cold. Heating.	Yellow ppt. Brown, resinous; partly sol.	No change. No change.	No change.
Alum. { Cold. Heating.	No change. No change.	Darker. Same.	Blue ppt. Blue ppt.
Potassium bichromate. { Cold. Heating.	Yellow ppt. Resinous; mostly dissolves.	No change. No change.	No change.
Ferric chloride. { Cold. Heating.	No change. Brown-yellow.		
Stannous chloride. { Cold. Heating.	No change. No change.	No change. No change.	Blue ppt.
Calcium hypochlorite. { Cold. Heating.	Dirty yellow ppt. Dirty red color.		
Zinc dust and ammonia. { Cold. Exposed. Precipitate.	Colorless. Colorless. Yellowish.	Colorless. Same.	Colorless. Blue.
Zinc dust and acetic acid. { Cold. Exposed. Precipitate.	Blue. No change.	Bluish pink. Same.	Colorless. Blue.

## SECTION XXV.

### MISCELLANEOUS TESTS IN DYEING.

**Experiment 160. The Amount of Dyestuff Necessary for a Full Shade.** — This factor may be determined by dyeing test-skeins with increasing amounts of dyestuff until no further increase in shade is observed. Proceed as follows: Use six test-skeins of woolen yarn (No. 1 to No. 6) of the same weight, dye them respectively with the following amounts of Acid Violet, employing the usual acid bath and method of dyeing:

No. 1, with 1 per cent. of dyestuff (436).

No. 2, with 2 per cent. of dyestuff (437).

No. 3, with 3 per cent. of dyestuff (438).

No. 4, with 4 per cent. of dyestuff (439).

No. 5, with 5 per cent. of dyestuff (440).

No. 6, with 6 per cent. of dyestuff (441).

After dyeing the samples are compared, and note is taken at which point the shade ceases to show a perceptible increase. The same method may be employed in other classes of dyes, using their respective methods of dyeing.

**Experiment 161. To Determine the Degree of Exhaustion of the Dye-bath.** — By the *exhaustion* of the dye-bath is meant the relative quantity of color absorbed by the fibre during the dyeing process. Proceed as follows: Dye a test-skein (442) of woolen yarn with 3 per cent. of Acid Violet, 4 per cent. of sulphuric acid, 20 per cent. of Glaubersalt, making the dye-bath up to exactly 300 cc. and taking out 25 cc. of the solution before dyeing to preserve for comparison in a test-tube. Carry out the dyeing operation in the usual manner; squeeze the excess of liquor from the skein back into the dye-bath so as not to lose any



of the solution. Make up the bath again to exactly 275 cc.; fill a graduated calorimetric tube with this solution; take 1 cc. of the original dye-bath in another similar calorimetric tube, and dilute the latter with water until it exhibits the same intensity of color as the first tube. The degree of dilution required measures the degree of exhaustion of the dye-bath. For example: 1 cc. of the original solution required to be diluted to 8.5 cc. to show the same intensity of color as the exhausted bath; hence the relative amount of dye left in the bath is  $1 \div 8.5 = 0.12$ , and the amount absorbed must have been 0.88 or 88 per cent. Therefore the degree of exhaustion in this case would be 88 per cent.

Pour the liquor taken from the exhausted bath back into the dye-bath, and without further addition of dyestuff or chemicals, dye another test-skein of woolen yarn. After dyeing a portion (443) compare it with the first skein. The difference in intensity of the two dyeings will represent in a rough manner the degree of exhaustion. Now continue the dyeing of the second skein (444) by adding to the bath sufficient dyestuff to bring the shade up to that of the first skein. Besides the dyestuff also add 2 per cent. more of sulphuric acid, as some of the acid originally added will have been removed by the first dyeing. Note the amount of dyestuff added, and this will represent the amount originally absorbed from the first bath. For example: it required the further addition of 2.5 per cent. of Acid Violet to match the second dyeing to the first; hence, the degree of exhaustion would be  $2.5 \div 3 \times 100 = 83.3$  per cent., which is a rather close approximation to the result obtained by the first method. The first method is the more accurate, but in some cases the bath after dyeing has a different color from that of starting, and, again, some dyes (mordant colors) give solutions which do not accurately represent the color obtained by dyeing; in which cases the latter method only could be used.

**Experiment 162. To Determine the Correct Amount of Mordant to Use.** — Mordant six test-skeins of woolen yarn each with 4 per cent. of tartar and the following amounts of chrome:

- |                        |                         |
|------------------------|-------------------------|
| (1) 1 per cent. (445). | (4) 5 per cent. (448).  |
| (2) 2 per cent. (446). | (5) 8 per cent. (449).  |
| (3) 3 per cent. (447). | (6) 12 per cent. (450). |

Enter at 140° F., raise to boiling, and mordant for 45 minutes; wash, and dye the skeins all together with 3 per cent. of Alizarin Red in the usual manner. Rinse and dry. Compare the several skeins, and by selecting the best color thus determine which percentage of chrome is the proper one to use. For nearly all purposes of mordanting it has been found that about 3 per cent. of chrome gives the best results. A larger amount of chrome appears to oxidize the wool and cause bad shades in dyeing; this is known as over-chroming and the wool becomes harsh and brittle.

**Experiment 163. To Determine the Degree of Exhaustion of the Mordant Bath.**—Mordant a test-skein of woolen yarn in a bath containing 300 cc. of water, 3 per cent. of chrome, 4 per cent. of tartar. Enter at 140° F., raise to boiling, and mordant for 45 minutes. Squeeze back the liquor from the skein into the bath and wash well. Add sufficient water to the mordant bath to bring its volume up to 300 cc. again, and without further additions, mordant a second skein in a similar manner. Repeat in the same way with a third skein. Then dye the three skeins together with 3 per cent. Alizarin Red in the usual manner, and finally compare the skeins for depth of shade in order to determine the relative exhaustion of the mordant bath (451, 452, 453).

Repeat this test, using a mordanting bath of 300 cc. of water, 3 per cent. of chrome, 2 per cent. of lactic acid, 2 per cent. of sulphuric acid. Mordant three test-skeins successively in the manner above described and dye again with 3 per cent. of Alizarin Red. Compare these (454, 455, 456) for depth of shade to determine the relative exhaustion of the bath and also to determine if the exhaustion is the same in the second case as in the first.

**Experiment 164. To Show the Dichroic Property of a Dyestuff.**—Coloring-matters are known as “dichroic” when they change their tone with change of intensity. Magenta, for instance, in

heavy shades is a red color, while in light tints it changes to a bluish pink. To show this property proceed as follows: Dye six tests of woolen yarn with Magenta, using 10 per cent. of glaubersalt in each dye-bath together with the following amounts of dyestuff:

- |                       |                          |
|-----------------------|--------------------------|
| (1) 3 per cent. (457) | (4) 0.5 per cent. (460)  |
| (2) 2 per cent. (458) | (5) 0.1 per cent. (461)  |
| (3) 1 per cent. (459) | (6) 0.01 per cent. (462) |

Enter at 100° F., raise to 180° F., and dye for 30 minutes. Observe the different tones in the colors obtained with change of concentration.

The dichroic nature of a coloring-matter may also be observed with its solution. Take a small quantity of Magenta and dissolve in 5 cc. of water in a test-tube; pour out half of this solution into a second test-tube and dilute with an equal quantity of water. Pour out half of the second solution into a third test-tube, and dilute again with four times the amount of water. Compare the colors of the three solutions, and notice the effect of dilution on the tone of the original color. In the same manner test the dichroic properties of Eosin, Methyl Violet, and Malachite Green.

**Experiment 165. Effect of Dichroism in the Compounding of Shades.** — Dye a test-skein of woolen yarn with 3 per cent. of Auramine (463), and another with 3 per cent. of Magenta (464). Then dye two more skeins, the one with  $\frac{1}{10}$  per cent. of Auramine (465), and the other with  $\frac{1}{10}$  per cent. of Magenta (466). Notice that the Auramine is not especially dichroic, whereas the Magenta is markedly so. Now dye a skein (467) with a mixture of 2 per cent. of Auramine and 3 per cent. of Magenta and another skein (468), with 2 per cent. of Auramine and 1 per cent. of Magenta and a third skein (469) with 2 per cent. of Auramine and  $\frac{1}{10}$  per cent. of Magenta. Notice the wide difference in the character of the colors obtained; for while the Magenta in the first case (3 per cent.) exercises the function of a red dye, in the last case ( $\frac{1}{10}$  per cent.) it acts as a violet dyestuff; hence the effect is entirely different in kind. Next dye a skein (470) with



$\frac{1}{10}$  per cent. of Auramine and  $\frac{1}{10}$  per cent. of Magenta, and another skein (471) with 2 per cent. of Auramine and 2 per cent. of Magenta. Theoretically, the first color should be a reduced tint of the second; but practically it will be seen that the two colors are of entirely different orders. This is caused by the wide variation in the color of the Magenta with different concentrations. From this it will be seen that the dichroic property of a dyestuff has an important bearing on its mixing qualities with other dyes in the compounding of shades. A red and a blue dye when used together in heavy shades may give a very satisfactory violet; but if the attempt is made to obtain a reduced tint of this violet by using small percentages of the two colors in the same proportions, it will perhaps be found that a tint of an entirely different color is obtained. The same is true when dealing with green and orange colors. In order to become acquainted with the true mixing qualities of his dyestuffs, the dyer should be familiar with the colors they give with large, medium and small percentages, and in the mixing of his shades he must make due allowance for the change in tone of color of the dyes with varying concentration.

#### SAMPLES.

- 436. With 1 per cent. of dyestuff.
- 437. With 2 per cent. of dyestuff.
- 438. With 3 per cent. of dyestuff.
- 439. With 4 per cent. of dyestuff.
- 440. With 5 per cent. of dyestuff.
- 441. With 6 per cent. of dyestuff.
- 442. Dyeing representing first bath.
- 443. Dyeing from exhausted bath.
- 444. Second dyeing matched to first.
- 445. With 1 per cent. of mordant.
- 446. With 2 per cent. of mordant.
- 447. With 3 per cent. of mordant.
- 448. With 5 per cent. of mordant.
- 449. With 8 per cent. of mordant.
- 450. With 12 per cent. of mordant.
- 451. Representing first bath of mordant.
- 452. Representing first exhaustion of mordant.

- 453. Representing second exhaustion of mordant.
- 454. First mordant bath with lactic acid.
- 455. First exhaustion with lactic acid.
- 456. Second exhaustion with lactic acid.
- 457-462. Dichroic property of Magenta.
- 463. Dyeing with 3 per cent. Auramine.
- 464. Dyeing with 3 per cent. Magenta.
- 465. Dyeing with  $\frac{1}{10}$  per cent. Auramine.
- 466. Dyeing with  $\frac{1}{10}$  per cent. Magenta.
- 467. Dyeing with 2 per cent. Auramine and 3 per cent. Magenta.
- 468. Dyeing with 2 per cent. Auramine and 1 per cent. Magenta.
- 469. Dyeing with 2 per cent. Auramine and  $\frac{1}{10}$  per cent. Magenta.
- 470. Dyeing with  $\frac{1}{10}$  per cent. Auramine and  $\frac{1}{10}$  per cent. Magenta.
- 471. Dyeing with 2 per cent. Auramine and 2 per cent. Magenta.

### QUIZ 25.

709. How would you determine the amount of dyestuff required to yield a full shade of color? What percentage of Acid Violet gives a satisfactory full shade?

710. What is meant by the degree of exhaustion of the dye-bath? How may this be determined? Give two methods.

711. If on a 2 per cent. dyeing for first bath it required 1.6 per cent. of dye for the second bath, what was the degree of exhaustion?

712. Describe the test to determine the correct amount of mordant to use. What percentage of chrome gave the best mordanting?

713. How may the degree of exhaustion of the mordant bath be determined? How does this compare for tartar and lactic acid assistants?

714. What is meant by the dichroic property of a dyestuff? What is the dichroism exhibited by Magenta?

715. How would you determine the dichroism of a dyestuff? What did you observe in the cases of Eosin, Methyl Violet, and Malachite Green?

716. What is the general effect of dichroism in the compounding of colors? How may this be shown?

## SECTION XXVI.

### TESTING THE FASTNESS OF COLORS.

IN Section IX, brief methods have already been given for the testing of dyed colors to various agencies. The present section is intended to be a more extended discussion of this subject.

By the "fastness" of a dye is meant its resistance to the action of various agencies to change it in color or appearance. The fastness of dyes differs very widely even among the same class; some acid dyes, for instance, are very fast while others are fugitive; and the same is true in general of the basic and substantive dyes. The mordant dyes are to be regarded as having the greatest fastness when taken as a class; and the basic dyes, as a class, are probably the most fugitive. Again, dyes may be fast to one agency and not to others; for instance, Thioflavin T dyed on cotton is very fast to washing and to fulling, but not at all fast to light. Further, a dye may be fast on one fibre and not on another; and may be fast when dyed by one method (or mordant) and not fast when dyed by another method (or mordant).

**Experiment 166. Testing Fastness of Colors Dyed on Wool.** — The chief agencies to which colors on wool are tested, together with the means of so testing them, are as follows:

(1) *Fastness to Light.* — A sample of the dyed wool is placed in a suitable frame in such a manner that only a part is exposed. The frame is then placed in such a position that it receives as strong sunlight as possible, but is shielded from exposure to the atmosphere by glass. A window with southern exposure is a good location in which to hang the frame containing the samples. At the end of one week's exposure the samples are examined and note made of those which show any appreciable fading; these are to be classified as **not fast**. At the end of the second week another examination is made and those samples noted which



show an appreciable fading; these are to be classified as **fairly fast**. At the end of four weeks the samples are once more examined and the colors fading in this period are noted and classified as **fast**. The samples which show no fading at the end of four weeks are classified as **very fast**.

Dye test-skeins of woolen yarn with 2 per cent. each of the ten following dyes, and test samples of the colors for fastness to light in the manner above described.

1. Magenta (in neutral bath).
2. Eosin (in acetic acid bath).
3. Acid Violet (in usual acid bath).
4. Tartrazin (in usual acid bath).
5. Patent Blue V (in usual acid bath).
6. Light Green SF (in usual acid bath).
7. Diamine Scarlet 3B (ammonium acetate bath).
8. Cloth Red GA (acid bath and after-chromed).
9. Alizarin Red WS (on alum mordant).
10. Alizarin Blue NG (on chrome mordant).

In making the test for fastness to light, as the nature and amount of sunlight obtainable day by day is very variable, a more accurate method is to expose, with the samples to be tested, control samples of dyes representing the four degrees of fastness to light, such as:

1. Very fast, Alizarin Red WS (on a chrome mordant).
2. Fast, Lanacyl Blue R (in usual acid bath).
3. Fairly fast, Alkali Blue R (in neutral bath and developed with acid).
4. Eosin (in weak acetic acid bath).

The samples are examined from time to time, and those colors fading in the same periods as the control samples are noted and classified accordingly. In this manner the variable effect due to the inconstant degree of light is eliminated, and the tests made comparable to certain fixed standards.

(2) *Fastness to Washing*. — This test is to represent the fastness of a dye to washing or scouring with soap and water.

Dyed woolen material of almost any character should be capable of standing a more or less severe scouring, as such an operation is always necessary in the cleansing and finishing of woolen goods in the course of their manufacture. Material dyed as loose stock must afterwards stand a rather severe scouring in order to remove the oils added for purposes of spinning; yarn-dyed material also accumulates considerable grease and dirt in handling and weaving and must also be scoured. Goods dyed in the piece are usually scoured before dyeing; hence colors in this latter case need not be especially fast to scouring, unless the character of the goods requires them to be subsequently scoured either in manufacturing or in wearing. The best manner of conducting the washing or scouring test is as follows: Plait together a few strands of the dyed test-skein with an equal portion of white wool yarn and white cotton yarn. Scour this sample for 10 minutes in a miniature scouring bath (about 50 cc.) containing 5 grams of soap per litre at a temperature of 140° F. Squeeze, wash off in fresh water, and dry. Note if the dye tints the soap solution, and if it tints either the white wool or the white cotton. The latter is used in the test as cotton threads are frequently employed in the weaving of woolen goods. Some dyes may tint the soap solution without staining the white yarns, but this may result in the staining of other colors, hence such dyes cannot be considered fast; again, some dyes may stain the white wool, and not the white cotton, or *vice versa*; in either case, the color must be classed as not fast. As to degrees of fastness, an arbitrary classification may be made as follows:

1. Fast; does not tint the soap liquor, nor either of the white yarns.
2. Fairly fast; tints the soap liquor, but not the white yarns.
3. Not fast; tints either of the white yarns; the soap liquor may or may not be tinted.

Make up plaited test samples from each of the ten colors given above, and test them to fastness to washing in the manner described; it is needless to add that a fresh portion of soap liquor must be used for each sample tested.

(3) *Fastness to Fulling.* — This is also called milling, and refers to the process whereby woolen cloth is felted more or less in order to make a denser fabric or to otherwise finish the goods. The felting is carried out in fulling mills or stocks, in which the material is saturated usually with an alkaline soap liquor and then rubbed and squeezed together until the desired degree of felting is obtained. The process of fulling is a very severe test on colors, and the mordant dyes are about the only ones which will stand a hard fulling; there are, however, certain acid and substantive colors on wool which will stand a fair degree of fulling. The basic dyes will not stand fulling.

The fulling test may best be carried out as follows:

Make a loose plait containing several strands of the dyed yarn mixed with strands of white woolen and cotton yarns, and treat with a solution containing 10 grams of soap and 2 grams of soda ash per litre at 140° F. Soak the sample in this solution and rub between two pieces of board until the wool yarns are well felted together. Then wash in fresh water, and dry. Note if the color has lost in intensity or if it has bled into either the white wool or cotton. In such case the dye cannot be considered fast to fulling. According to the degree of bleeding, the color may be classed as **not fast** or as **fairly fast**. If the dye neither loses in color nor bleeds, it may be classed as **fast**. Prepare test samples from each of the ten dyes given and test them in manner described for fastness to fulling.

(4) *Fastness to Rubbing.* — This is also termed "crocking," and refers to whether or not the dye will mechanically rub off, and thus stain white or other colors with which it may come in contact. Heavy shades are more apt to rub than light shades. As a rule, the acid and substantive dyes on wool do not rub; the basic dyes frequently show this defect; heavy shades of mordant (or pigment dyes in general) will frequently rub off to some extent, whereas lighter shades do not. Heavy shades of indigo (a pigment dye), for instance, rub off considerably. The test for fastness to rubbing is easily and simply carried out by rubbing a portion of the dyed sample on a piece of white calico, and



noting if a stain is left. Test the ten dyed samples in this manner, and classify as **fast** or **not fast** to rubbing.

(5) *Fastness to Water*. — The object of this is to discover if the dye will bleed into white yarn on boiling in water or on prolonged steeping in cold water. Test as follows: (a) Plait several strands of the dyed yarn with some white wool and white cotton yarns, and boil with water for 1 hour. Squeeze and dry. Note if the white yarns become stained. (b) Use another plaited sample as above, and steep in cold water for 12 hours. Note if the white yarns become stained. If the dye does not bleed at all in boiling water it may be classed as **fast**; if it bleeds slightly in boiling water, but not in cold water, it is **fairly fast**; and if it bleeds in the cold water test, it is **not fast**. Carry out tests on the ten dyed samples in the manner described, and classify these dyes with respect to their fastness to water.

(6) *Fastness to Weather*. — By this is meant fastness to the varying conditions of exposure to the atmosphere, such as alternate wetting by rain or dew and drying by the heat of the sun, etc. The best and most practical method of applying this test is to expose a sample of the dyed material to the action of the weather for two weeks or more. But the results may be approximately represented by the following test. (a) Steep a sample of the dyed yarn in a solution containing 2 parts of hydrogen peroxide (10-volume strength) and 10 parts of water for 1 hour. Dry and compare with the original sample. (b) Repeat the test, using a hydrogen peroxide solution of 10-volume strength undiluted with water; steep for 1 hour, and on drying compare with the original sample. If no alteration in the color is appreciable after test (b), the dye may be classed as **fast**; if test (b) shows an alteration in the color, but not test (a), the dye may be classed as **fairly fast**; if test (a) shows an appreciable alteration, the dye is **not fast**. By combining this test with the one to sunlight, a fair idea of the fastness of the dye to weather exposure may be gained. It is probable that in the wetting of material by rain or dew and subsequent evaporation by the heat of the sun, a trace of hydrogen peroxide is formed which has a bleaching action on colors.

(7) *Fastness to Acids and Carbonizing.* — In many cases dyed woolen materials are treated with moderately strong solutions of acids and dried in order to decompose any particles of vegetable matter which may be present; this process is known as carbonizing. To test the fastness of a dyestuff to this operation, proceed as follows: Immerse a sample of the dyed yarn in a solution of sulphuric acid of 4° Tw. at 175° F. for half an hour; squeeze and without washing dry in a hot air flue. Then wash out and neutralize the acid in a bath containing about 1 gram of soda ash to 100 cc. of water; finally rinse well, and dry. Compare with the original color, and note if the carbonizing process has altered the shade in any manner. According to the extent of change in the shade classify as **not fast**, **fairly fast**, and **fast**. Test in this manner each of the ten dyed samples.

(8) *Fastness to Perspiration.* — This is required of all dyed clothing material that is worn next the skin; also of material used for making horse-blankets, etc. The most reliable test is to wear a sample of the dyed wool in such a manner as to expose it to the action of perspiration. This action, however, may be well represented by the following test: Plait a sample of the dyed yarn with white woolen and cotton yarns, and immerse for 1 hour in a solution of acetic acid of 4° Tw. at 100° F. Squeeze, and dry without washing in the air. Note if the color has suffered any alteration in shade or if it has stained either of the white yarns. According to the extent of change or staining classify as **not fast**, **fairly fast**, or **fast**.

(9) *Fastness to Alkali.* — In order to remove the fatty matters from woolen goods a washing with dilute soda ash solution is frequently given. This is especially true of material which is fulled. To discover if the color will withstand such a treatment, a test is made as follows: A sample of the dyed yarn is plaited with white wool and white cotton, and steeped for one hour in a solution of soda ash of 3° Tw. at 120° F., then washed in fresh water and dried. Note if the color suffers any alteration, or if either of the white yarns is stained. According to the extent of

change in color or staining the dye is to be classified as **not fast**, **fairly fast**, or **fast**.

(10) *Fastness to Lime or Street Dust.* — Dyed clothing materials such as ladies' dress goods and gentlemen's suitings have to withstand the action of street dust, mud, etc. This action is best represented by a test with lime as follows: Spot a sample of the dyed yarn with a solution containing 20 grams quicklime and 10 cc. ammonia per litre. Allow this to dry on the material, and then brush off. Note if the color has suffered any alteration.

(11) *Fastness to Sulphuring or Stoving.* — In some cases dyed woolen yarn is woven together with white and the cloth subsequently bleached by the action of sulphurous acid gas. To discover if the dye will withstand the action of the sulphurous acid test as follows: Take a small sample of the dyed yarn, moisten it with water, and hang it for 6 hours in a closed bottle filled with sulphurous acid gas (obtained by burning a piece of sulphur in the bottle). Note if the color undergoes any alteration, and corresponding to the extent of change classify the dye as **not fast**, **fairly fast**, or **fast**.

(12) *Fastness to Steaming.* — In the various finishing operations, dyed woolen fabrics may be subjected to a steaming operation in order to give the surface of the goods a lustre and a certain finish. This process is frequently called "decatizing" or "pott-ing." The same operation is carried out on goods composed of wool and cotton yarns in order to prevent crinkling, in which case it is called "crabbing." To test a dyestuff to the influence of such an operation proceed as follows: Prepare a plaited sample containing the dyed yarn together with white wool and cotton. Steam the sample for one-half hour under about 5 pounds pressure. Note if the color suffers any alteration, or if it stains the white yarns. According to the extent of change or staining classify the dye as **not fast**, **fairly fast**, or **fast**.

(13) *Fastness to Hot Pressing or Ironing.* — Woolen material employed in the manufacture of suitings, etc., requires to be hot pressed or ironed. To discover if a dyestuff will withstand such a treatment, test as follows: (a) Moisten a sample of the dyed



yarn and press with a hot iron till dry. Note if the color undergoes any alteration on cooling. (b) Moisten a sample of the dyed yarn and cover with a piece of white muslin, then press with a hot iron until dry. Note if the color suffers any alteration or if it stains the white muslin. If no change takes place under (a) class the dye as **fast**; if it changes under (a) but not under (b), or stains the white slightly without any other perceptible change, class as **fairly fast**; if the color is altered by both (a) and (b) class as **not fast**.

**Experiment 167. Testing the Fastness of Cotton Dyeings.** — In many cases dyeings on cotton materials are tested in the same manner as on wool; but there are deviations from the latter, owing to the different character of the fibre. The following are the chief tests to be applied to dyed cotton material:

(1) *Fastness to Light.* — Tested in the same manner as with wool.

(2) *Fastness to Washing.* — Tested in the same manner as with wool.

(3) *Fastness to Fulling.* — Tested in the same manner as with wool.

(4) *Fastness to Rubbing.* — Tested in the same manner as with wool.

(5) *Fastness to Water.* — Tested in the same manner as with wool.

(6) *Fastness to Acid.* — It is frequently the practice to weave dyed cotton yarn with white wool and subsequently to dye the wool with acid colors. This is termed "cross-dyeing." To discover if the dyed cotton will withstand the action of the boiling acid dye-bath, test as follows: Plait a sample of the dyed yarn with white wool and cotton, and boil for 1 hour in a solution containing 1 cc. sulphuric acid and 2.5 grams Glaubersalt per litre. Then wash and dry. Note if the color sustains any alteration or if it bleeds into the white yarns, and classify the dye as **not fast**, **fairly fast**, or **fast**.

(7) *Fastness to Weather.* — Tested with hydrogen peroxide in the same manner as with wool.

(8) *Fastness to Perspiration*. — Tested with acetic acid in the same manner as with wool.

(9) *Fastness to Alkali*. — This is tested in the following manner: (a) Steep a sample of the dyed yarn for 2 minutes in cold ammonia water (full strength), and observe if the color undergoes any alteration. (b) Steep a similar sample in a solution containing 10 grams of soda ash to 100 cc. of water for 2 minutes, and dry without washing. Note if the color undergoes any alteration. (c) Plait a sample of the dyed yarn with white wool and cotton, and boil for one-half hour in a solution containing 2 grams of soda ash per litre; rinse and dry. Observe if the color suffers any change, and if the white yarns become tinted. From these tests classify the color as **fast**, **fairly fast**, or **not fast**.

(10) *Fastness to Mercerizing*. — In some cases cotton is mercerized after being dyed; and as the mercerizing process consists in treating the material with strong solutions of caustic soda, it is necessary that the dyed color should not be affected by this treatment in order to be employed on this class of material. Carry out the test as follows: Steep a sample of the dyed yarn in a solution of caustic soda of 50° Tw. for 5 minutes; wash well in cold water, then in hot water, and finally in water acidulated with acetic acid; dry, and observe if the color has undergone any alteration, and classify accordingly as **fast**, **fairly fast**, or **not fast**.

(11) *Fastness to Chlorine or Bleaching*. — Cotton fabrics containing white interwoven with colored yarns are frequently bleached more or less thoroughly in order to clear the white; such material as cotton towelling containing colored borders is also bleached quite thoroughly. To discover if a dye will withstand the action of bleaching which is done with solutions of chloride of lime, the following test should be made: Steep a sample of the dyed yarn in a cold solution of chloride of lime of 1½° Tw. for 1 hour. Rinse in water slightly acidulated with hydrochloric acid, then in dilute soap solution, and finally dry. Observe if the color has undergone any alteration in shade, and classify accordingly as **fast**, **fairly fast**, and **not fast**.

(12) *Fastness to Lime or Street Dust.* — This is determined in the same manner as with wool.

(13) *Fastness to Steaming.* — This is determined in the same manner as with wool.

(14) *Fastness to Ironing or Hot Pressing.* — This is determined in the same manner as with wool.

Dye ten test-skeins of cotton with 2 per cent. of the following dyestuffs, and test each color with respect to fastness to the different agencies as above described:

1. Benzopurpurine (substantive dye).
2. Chrysophenine (substantive dye).
3. Diamine Blue RW (substantive dye after-treated with bluestone).
4. Dianil Direct Yellow S (substantive dye).
5. Dianil Brown 3GO (substantive dye after-treated with chrome).
6. Methyl Violet (basic dye on tannin-antimony mordant).
7. Magenta (basic dye on tannin-antimony mordant).
8. Methylene Blue (basic dye on tannin-antimony mordant).
9. Cotton Blue (acid dye on "blue mordant").
10. Rhodamine (basic dye on oil-aluminium mordant).

#### TABULATION OF FASTNESS REQUIRED ON VARIOUS CLASSES OF MATERIALS.

##### (a) Wool.

(1) *Loose Wool.* — Fastness to light, fulling, and potting; the dyes recommended are mordant colors first, and substantive dyes next. The latter are quite fast to scouring on wool, and fairly fast to fulling; they are especially recommended for dyeing in machines on account of their good solubility.

(2) *Shoddy, Mungo, and Carbonized Rags.* — Fastness to fulling and cheapness in dyeing; fastness to light is seldom required. The dyes recommended are the mordant and substantive colors in the first place, and secondly the basic colors. The latter are only fast to very light fulling, but do not stain white cotton, and are valuable on account of their great brilliancy.





[illegible]

(3) *Slubbing and Tops*. — The fastness required and the dyes recommended are the same as for loose wool.

(4) *Weaving Yarns (Worsted, Cheviot and Carded Yarns)*. — As these are chiefly used for scouring and fulling purposes, the fastness required and the dyes recommended are the same as for loose wool.

(5) *Worsted Knitting Yarns and Hosiery Yarns*. — These require fastness to scouring, perspiration, and rubbing. Substantive and mordant dyes are recommended; also the faster acid dyes.

(6) *Yarns for Flannels, Rugs, Blankets, Plaids, etc.* — These require a moderate fastness to fulling; also fastness to perspiration and rubbing, and for rug yarns fastness to stoving is frequently desired. Substantive and mordant dyes are recommended as well as the faster acid colors, especially those which are after-treated.

(7) *Carpet and Tapestry Yarns*. — These require fastness to light, cold water, and rubbing; dyes possessed of good levelling properties are also desired. The best dyes are acid colors fast to light, then the mordant dyes, and the substantive dyes which are after-treated with bluestone.

(8) *Worsted Braids*. — These require dyes that penetrate well, and that are fast to light and rubbing. The same colors as for the preceding are recommended.

(9) *Fancy Yarns*. — Fastness to stoving and clear brilliant colors are usually desired. The best dyes to use are the level dyeing acid colors and the basic colors.

(10) *Piece Goods consisting of Woolen Cloths (Beavers and Meltons), Gentlemen's Suitings, Worsted Coatings, Cheviots, Carded Woolen Cloths, Braids for Army and Navy Cloths, Billiard Cloth*. — In such goods it is desirable that the dyes be level and penetrate well; fastness to light, potting, hot pressing, and rubbing is also required, and in some cases, fastness to carbonizing. The dyes best to use are the faster acid colors and the mordant colors, and secondly the substantive dyes.

(11) *Dress Goods, Ladies' Cloths, Dogskins, Doeskins, Cashmeres, Crêpons, etc.* — These require the same fastness as the



preceding, and also fastness to street dust. The same dyes are used as with the preceding.

(12) *Flannels*. — These principally require fastness to washing and light; the dyes chiefly used are the acid and substantive colors, the latter being largely employed for red shades.

(13) *Velvets and Plushes*. — These require fastness to light and rubbing; acid dyes are principally used.

(14) *Woolen Felt*. — Good penetration and level dyeing is the first consideration; the fastness required will vary with the particular use to which the goods are put. The dyes mostly used are the easily soluble acid colors and the substantive colors.

(15) *Hats of Wool and Hair*. — In this case fastness to light, potting, and rubbing is required; also the color must penetrate well. The dyes principally used are mordant colors, and readily soluble acid colors fast to light.

#### (b) Cotton.

(1) *Loose Cotton and Slubbing*. — These require to be fast to fulling and light, though the requirements vary greatly, according to the use to which the material is to be put. The dyes mostly used are the substantive colors, dyed direct, diazotized and developed, or after-treated; also substantive dyes topped with basic colors, and even basic colors alone.

(2) *Fancy Weaving Yarns, Cotton Warps for Union Goods, Knitting Yarns, Hosiery Yarns*. — These require fastness to scouring (and sometimes fulling) to cross-dyeing, to light, and generally to perspiration, though the degree of fastness will necessarily vary considerably with the use to which the yarn is to be put. The dyes recommended are the diazotized and developed and the after-treated substantive colors, sulphur colors, basic colors, and substantive colors topped with basic; for light shades, the direct dyed substantive colors. For cop dyeing the substantive colors of ready solubility are principally used.

(3) *Yarns for Draperies, Upholstery, etc.* — These chiefly require fastness to light and rubbing. The dyes mostly used are the substantive colors, the basic colors, and some of the acid colors.

(4) *Sewing Cotton*. — This requires fastness to light and rubbing. The substantive and basic dyes are chiefly used.

(5) *Cotton Hosiery and Knit Goods*. — These require fastness to washing, perspiration, rubbing, and sometimes light. The dyes must also penetrate well. The colors used are the same as for knitting yarns; also aniline black and the sulphur colors.

(6) *Piece Goods consisting of Moleskins, Cotton Worsteds, Beavers, Fustians, Flannelettes, Sateens, Plushes, Velvets, Corduroys, etc.* — These usually require fastness to perspiration, light, rubbing, hot pressing, and in some cases also fastness to washing. The dyes mostly used are the substantive colors dyed direct, also diazotized and developed, and after-treated; also the sulphur dyes; also substantive dyes topped with basic colors, and at times basic dyes alone.

(7) *Cotton Linings, Bobbinet, Tulle, etc.* — These require fastness to rubbing, perspiration, and frequently hot pressing. The dyes chiefly used are the substantive colors dyed direct, diazotized and developed, or topped with basic dyes; the sulphur dyes; and the basic dyes. Bobbinet is frequently dyed in the sizing.

(8) *Bookbinders' Cloth*. — About the only fastness required is to light. The dyeing is frequently done in the sizing, using acid dyes, basic dyes, and substantive dyes.

#### (c) Union Goods.

(1) *Thread Waste, Shoddy, etc.* — Moderate fastness to fulling is usually required together with cheapness of dyeing. The dyes mostly used are the substantive colors, either alone or in conjunction with neutral dyeing acid colors; also after-treated substantive colors.

(2) *Merino and Angola Yarns, Braids, etc.* — The requirements for fastness vary with the application. The dyes used are those given above, also substantive dyes topped with basic colors.

(3) *Hosiery Yarns*. — These require fastness to perspiration, washing, and rubbing. The dyes employed are those given above.

(4) *Piece Goods consisting of Cotton Warp Suitings, Woolens, Dress Goods, Shawls, Crewels, Astrachans, Italian Cloth, Serges, Hosiery, Felt, and Flannels.* — The requirements for fastness vary with the nature and use of the material; in general, fastness to washing and rubbing is desired; fastness to light is of minor importance with linings and hosiery. The dyes mostly used are the substantive dyes either alone or in combination with the neutral dyeing acid colors.

(5) *Ladies' Cloths, Presidents, Whitneys, Moscows, Beavers, Worsted Coatings, etc.* — The only important requirement is sufficient fastness to light, water, rubbing, and hot pressing. The dye should cover the cotton well, be level dyeing, and have good penetration. The substantive dyes are principally used, either alone or in combination with neutral dyeing acid colors.

#### QUIZ 26.

717. What is meant by the "fastness" of a dyestuff? Is this fastness a constant factor among different dyes of the same class? Which class of dyes is to be regarded as the fastest? Which most fugitive?

718. Does the same dye exhibit the same degree of fastness on different fibres? Have different methods of dyeing any influence on the fastness of the colors?

719. What are the agencies to which dyes on woolen materials are tested?

720. Give the method of testing fastness to light. Should exposure to the atmosphere be included in a light test, and why?

721. Why are standards used in making the light test, and what are these standards?

722. What does the washing test represent? Is woolen material always scoured after dyeing? Which class of goods should have dyes fast to washing, loose stock, yarn, or cloth?

723. Describe the washing or scouring test. Why is the sample mixed with white wool and cotton?

724. How are dyes classified as to degrees of fastness to washing?

725. What is meant by "fulling"? By what other term is it known? Is this process severe on colors? Which class of dyes is fastest to fulling?

726. Describe the method of testing fastness to fulling.

727. What is meant by the "crocking" test? How is it carried out?

728. What is understood by fastness to "water"? How is the test made?



729. What is meant by fastness to "weather"? How may the test be represented by chemical means? With what other test should this always be combined?

730. What is "carbonizing"? How is the test made?

731. Give the best method of making the perspiration test. By what chemical test may it be represented?

732. In what case is a treatment with alkali given dyed woolen fabrics? How is the test for fastness to alkali conducted?

733. How is fastness to street dust represented by chemical means?

734. What is meant by "stoving"? Under what circumstances would a color be required to stand this test? How is the test for fastness made?

735. What is "decatizing," "crabbing"? How may the fastness of a color to steaming be tested?

736. The colors on what classes of materials require fastness to ironing or hot-pressing? How is this test made?

737. In what respects does the testing of dyed colors on cotton differ from those on wool?

738. How is the test for fastness to acid carried out? What is meant by "cross-dyeing"?

739. How does the test for fastness to alkali differ on woolen and on cotton goods?

740. What is meant by "mercerizing"? Under what conditions would this fastness be required, and how is the test made?

741. How does the bleaching test for cotton colors differ from that of stoving?

742. Referring to the cotton colors tested, how do the basic dyes compare in general fastness with the substantive dyes? In what particulars do the latter surpass the basic dyes?

743. What fastness is required on loose wool dyeings, and what are the chief classes of colors used on this material? What character of dyes is used or machine dyeing?

744. What fastness is required for shoddy dyeings? Why are basic dyes used largely?

745. What are slubbing and tops? What fastness is required for these dyeings?

746. What fastness is generally necessary for worsted and woolen weaving yarns? What dyes are mostly used?

747. What fastness is required for knitting and hosiery yarns of wool? Yarns for rugs, blankets, and flannels?

748. What fastness is required on carpet and tapestry yarns? Yarns for worsted braid?

749. What are the dyes employed for woolen piece dyeings? What is meant by fastness to "potting"?

750. What fastness is required on ladies' dress goods? What is the chief fastness for flannels?

751. What fastness is required for velvets and plushes? What dyes are chiefly used? What qualities are necessary for colors on woolen felts?

752. What fastness is required on loose cotton and slubbing? On cotton warps for union goods? For cotton hosiery yarns?

753. For cop-dyed cotton yarns what colors are best to use and why?

754. Colors on cotton draperies and upholstery require what fastness? What dyes are chiefly used on sewing cotton?

755. What fastness is required for colors on the general class of cotton piece goods? Cotton linings? What is meant by dyeing in the sizing?

756. For bookbinders' cloth what fastness is desirable? What kind of dyes are mostly used?

757. What are "merino" and "angola" yarns? What fastness is required on these? What dyes are used?

758. Name some of the principal varieties of union clothing material. What fastness is desirable for such colors?

## SECTION XXVII.

### ANALYSIS OF TEXTILE FABRICS.

**Experiment 168. To Determine the Amount of Wool and Cotton in a Fabric.** — A weighed portion of the sample is boiled for 10 minutes in a 5 per cent. solution of caustic potash, then washed well first with fresh water and afterwards with water slightly acidulated with acetic acid to remove all trace of alkali. The residue is dried and weighed. As the wool is dissolved by the alkali, the loss in weight corresponds to the amount of wool, while the residue represents the cotton. Record the results as in the following example:

Weight of sample.....	5.25 grams.
After boiling in caustic potash.....	1.06 grams.
Loss equals wool.....	4.19 grams.
Residue equals cotton.....	1.06 grams.

Hence:                      Wool = 79.8 per cent.  
                                Cotton = 20.2 per cent.

As the cotton present will suffer a slight loss in the process, it is customary to add 5 per cent. of its weight to the cotton, and to subtract this amount from that of the wool. With this correction the above figures become:

Wool = 78.79 per cent.  
Cotton = 21.21 per cent.

**Experiment 169. Analysis of Fabric Containing Silk and Cotton.** — (a) *Nickel Hydrate Method.* A weighed portion of the fabric (about 5 grams) is steeped for 5 minutes in a cold solution of nickel hydrate in ammonia; then heated almost to boiling for 5 minutes. This treatment should dissolve the silk completely. The residue of cotton is thoroughly washed, dried,



and weighed. The nickel hydrate solution for this test is prepared as follows: 25 grams of crystallized nickel sulphate are dissolved in 500 cc. of water; then sufficient caustic soda solution is added to completely precipitate the nickel as hydrate. This precipitate is well washed with water by settling and decantation, and finally rinsed into a 250 cc. flask with 125 cc. of water. The flask is next filled with ammonia water and well shaken, and the nickel hydrate should finally completely dissolve.

(b) *Zinc Chloride Method.* — A weighed portion of the sample is boiled for 2 minutes in a solution of basic zinc chloride of 1.72 sp. gr. The residue of cotton is thoroughly washed first with dilute hydrochloric acid, and then with water, and then dried and weighed. This treatment dissolves the silk without materially affecting the cotton. The basic zinc chloride solution is prepared as follows: 100 grams of zinc chloride and 4 grams of zinc oxide are dissolved in 85 cc. of hot water; after complete solution the liquid should have a density of 1.72. This method of analysis is well adapted for plushes and other heavy silk fabrics.

**Experiment 170. Analysis of Fabric Containing Wool and Silk.** — A weighed portion of the sample is steeped for 2 minutes in concentrated hydrochloric acid at 120° F. This will dissolve the silk without materially affecting the wool. Wash the residue of wool, dry, and reweigh.

**Experiment 171. Analysis of Fabric Containing Wool, Silk, and Cotton.** — A weighed portion of the sample is treated for 10 minutes with a cold solution of nickel hydrate in ammonia (see above). This will dissolve any silk present. Wash well, dry, and reweigh. The loss in weight represents *silk*. The residue is next boiled for 10 minutes in a 5 per cent. solution of caustic potash. This will dissolve any wool present. Wash well, dry, and reweigh. The loss in weight represents *wool*, while the residue consists of *cotton* (see Exp. 167 for correction to apply to weight of cotton).

**Experiment 172. Distinction between True Silk and Artificial Silk.** — Artificial silk (or *lustra-cellulose*) is a fibre prepared

from a solution of collodion or other cellulose solution. It consists of cellulose, whereas true silk is a nitrogenous animal substance. Artificial silk burns readily in the air like cotton, without evolving a strong odor; while silk burns slowly, and emits a characteristic odor. To estimate the amount of artificial silk present in a mixed fabric, a weighed portion of the sample is treated at the ordinary temperature for 20 minutes with an alkaline solution of copper sulphate. This will completely dissolve the natural silk, leaving the artificial fibre as a residue. The latter is thoroughly washed, dried, and reweighed. The alkaline solution of copper sulphate is prepared by dissolving 10 grams of copper sulphate in 100 cc. of water and 5 cc. of glycerin; a strong solution of caustic soda is then added until the precipitate at first formed just redissolves.

**Experiment 173. To Distinguish between Cotton and Linen. —**

(a) Steep the sample containing these two fibres for 2 minutes in concentrated sulphuric acid; wash well with water, gently rub with the fingers, and finally steep in dilute ammonia; then squeeze and dry. The cotton fibres will be converted into a jelly-like mass by the action of the acid, and is more or less completely removed by the rubbing and washing. The linen remains but little altered. By weighing the sample before and after the treatment an approximate idea of the amounts of cotton and linen present may be obtained. (b) Steep the sample to be tested in olive oil; then press between filter paper to remove the excess of oil. The linen fibres will become gelatinous in appearance and translucent, whereas the cotton remains unaltered. When placed on a dark background the linen fibres will now appear dark and the cotton fibres light. (c) Steep the sample to be tested in an alcoholic solution of rosolic acid, and then in a strong solution of caustic soda; finally rinse in water. The linen fibres will become rose-colored, while the cotton is colored much lighter and most of the color is removed by the rinsing. None of these tests are very satisfactory when the linen has been bleached for then its cellulose is practically identical with that of cotton. The most satisfactory means of qualitatively distinguishing linen

from cotton is by a microscopic examination, as these fibres exhibit very different microscopic properties (see the author's *Textile Fibres*).

**Experiment 174. To Distinguish between True Silk and Tussah Silk.** — Tussah silk (and the wild silks in general) may be distinguished from true silk by the following reactions:

(a) Tussah silk is only partially dissolved by cold concentrated hydrochloric acid (sp. gr. 1.16), even on standing for 48 hours; whereas true silk dissolves almost instantly.

(b) Tussah silk requires a comparatively long time to dissolve in the solution of basic zinc chloride, mentioned in Exp. 168; whereas true silk dissolves quite readily.

(c) True silk dissolves completely in a semi-saturated solution of chromic acid when boiled for 1 minute; whereas tussah silk remains unaltered after boiling for 2 to 3 minutes in this solution.

(d) To estimate the amount of tussah silk in a fabric, weigh off a portion of the sample and steep for 10 minutes in cold concentrated hydrochloric acid; wash the residue thoroughly, dry, and reweigh. The loss in weight represents the true silk while the residue consists of tussah silk.

### QUIZ 27.

759. Describe the method for determining the amounts of wool and cotton in a fabric.

760. Why is a correction applied to the quantity of cotton, and what does this correction amount to?

761. Give two methods for determining the amounts of silk and cotton in a fabric.

762. How is the ammoniacal nickel hydrate solution prepared? Which fibre is soluble in this reagent?

763. Give the method of preparing the solution of basic zinc chloride. Which fibre is soluble in this reagent?

764. If a fabric contains wool and silk, what method would you employ to determine the amount of each?

765. A fabric contains wool, silk, and cotton fibres; how would you determine the amount of each fibre present?



766. Of what does artificial silk consist? How may this fibre be qualitatively distinguished from true silk?

767. Give a method for determining the amount of artificial silk in a fabric.

768. Describe several methods for distinguishing between cotton and linen fibres in a fabric. Will bleached linen react in a similar manner?

769. Give several reactions to distinguish between tussah silk and true silk. What is the difference in origin between tussah silk and true silk?

770. How would you estimate the amounts of tussah silk and true silk in a fabric?

## SECTION XXVIII.

### ANALYSIS OF TEXTILE FABRICS.

**Experiment 175. Estimation of Sizing and Dressing Materials in a Fabric.** — These materials include *sizing*, such as **starch**, **clay**, etc., used for stiffening a warp or fabric; *finishing materials*, such as **glue**, **magnesium chloride**, etc., which may be added to give a certain finish to the cloth; **mordants** and **dyestuffs**, as well as **grease**, etc., which may be present in the fabric. A weighed sample of the fabric is boiled for 15 minutes in a 3 per cent. solution of hydrochloric acid; wash well, and boil for 10 minutes in a 1 per cent. solution of soda ash; wash well again, and dry. Reweigh, and the loss will represent the amount of size and dressing materials.

**Experiment 176. Conditioning of Textile Materials.** — By “conditioning” is meant the estimation of the amount of moisture present in a yarn or fabric and the subsequent calculation of the amount of normally dry fibre present in the sample. The test is usually carried out by means of a specially constructed conditioning oven wherein the weighed sample is heated at about 220° F. until the moisture is completely driven out. The residue consists of “bone-dry” fibre, and the loss is moisture. To the weight of the dry residue is then added the amount of moisture corresponding to that normally present in the fibre under examination. This amount is termed the “regain,” and though the standard varies in different localities, for America it may be taken as follows:

For materials of wool.....	18 per cent.
For materials of cotton.....	8½ per cent.
For silk.....	11 per cent.

Where a special conditioning oven is not available the moisture test may be made on a small sample contained in a weighing

bottle by heating in an ordinary drying oven. The test is much more accurate, however, when a relatively large amount of material is used.

Place 100 grams of woolen yarn in the conditioning oven and heat at 220° F. until no further loss in weight occurs. This will usually require 3 to 4 hours. Note the loss in weight. For example, suppose this loss to be 19.6 grams; then

Original weight.....	100.	grams.
Loss as moisture.....	<u>19.6</u>	grams.
Residue as bone-dry fibre.....	80.4	grams.
Add regain, 18 per cent. of this.....	<u>14.47</u>	grams.
Normal weight.....	94.87	grams.

Hence, the sample contained 94.87 per cent. of "conditioned" or normal wool.

Repeat the test, using samples of loose wool, worsted tops, raw cotton, cotton yarn, raw silk, etc., and calculate the amounts of conditioned fibre in each case.

**Experiment 177. Estimation of Oil and Grease in Fabrics.** — For analytical purposes, these substances are best extracted by means of petroleum ether (ligroin) in a Soxhlet extraction apparatus. A weighed quantity (about 1 gram) of the fabric to be tested is placed in the capsule of the extractor; pour 60 cc. of petroleum ether in the tared flask of the apparatus. Connect the extractor with its condenser and heat on a water-bath, regulating the temperature so that the solvent siphons over about every 5 minutes. Continue the extraction for 1 hour; then disconnect the extractor, distil off the solvent, dry the flask in a water oven, cool and finally reweigh. The increase in weight of the flask will represent the amount of grease and oil in the fabric.

**Experiment 178. Detection of Mineral Oil in Textile Fabrics.** — This oil is sometimes employed for the oiling of stock, and is sometimes difficult to remove by simple scouring, if such oil is of improper quality. Extract about 10 grams of the sample with 50 cc. of carbon tetrachloride in a Soxhlet extractor. Distil off the solvent; mix the residue of grease and oil with water. If



mineral oil is present a fluorescence will be noticed on the surface of the water.

**Experiment 179. Detection of Rosin Oil in Textile Fabrics. —**

A weighed portion (about 5 grams) of the sample is extracted with 50 cc. of ligroin (as in Exp. 176). To the residue of grease, after evaporation of the solvent, add 5 cc. of acetic anhydride; shake well at a gentle heat, then allow to cool, draw off the layer of acetic anhydride with a pipette, and add to it a drop of concentrated sulphuric acid. The production of a violet color (which, however, is not permanent) will indicate the presence of resin oils. If the presence of resin (colophony) in a textile fabric is suspected, the former may be first converted into resin oils by boiling with dilute hydrochloric acid; after which the test is to be carried out as given.

**Experiment 180. Estimation of Mineral Matter in a Fabric. —**

A weighed portion (1 to 2 grams) of the sample is clipped up and ignited in a tared porcelain crucible to a complete ash. The weight of the latter represents the amount of mineral matter in the sample.

**Experiment 181. Determination of the Nature of Sizing on a Fabric. —** The principal ingredients liable to be present in the sizing on a fabric are starch, dextrin, gums, gelatin, Irish moss, sugar, resin, fatty matters, and various inorganic substances such as china clay, gypsum, talc, magnesia, magnesium chloride, calcium chloride, zinc chloride, alumina, etc.

To test for these substances take a sample of the fabric measuring about 30 to 40 square inches, and boil for several hours in about 250 cc. of water. This will dissolve all soluble materials, including starch, dextrin, gums, gelatin, Irish moss, sugar, and the chlorides of magnesium, calcium, and zinc. Test this aqueous extract in the following manner:

(1) *Starch.* — Add to a portion of the solution a few drops of iodine solution (in potassium iodide); the formation of a blue color indicates the presence of starch.

(2) *Dextrin and gums.* — If no starch is present, concentrate a portion of the extract by boiling; cool, and add three times its

volume of alcohol. Dextrin or gums, if present, will be precipitated. These may be filtered off and identified by burning.

(3) *Gelatin*. — To a portion of the extract add some tannic acid solution, which will give a precipitate in the presence of gelatin.

(4) *Sugar and glucose*. — These may be detected by boiling a portion of the extract with a little dilute hydrochloric acid, and adding a few drops of Fehling's solution, when a red precipitate of cuprous oxide will be formed in the presence of a sugar.

(5) *Irish moss* (or other lichen jelly) may be considered to be present if an organic substance is known to exist in the extract, and yet no evidence of the foregoing compounds is found.

(6) *Chlorides* may be detected by adding a few drops of nitric acid to the extract, followed by a few drops of a solution of silver nitrate; the formation of a white precipitate of silver chloride will show the presence of chlorides.

(7) *Zinc* may be detected by adding ammonium sulphide to the extract; a white precipitate of zinc sulphide will indicate the presence of this metal.

(8) *Calcium* may be detected by adding ammonium oxalate solution to a portion of the extract, when a white precipitate of calcium oxalate will be formed in the presence of calcium compounds.

(9) *Magnesium* may be detected by adding a few drops of ammonia water and ammonium chloride solution to a portion of the extract, followed by the addition of sodium phosphate solution. The formation of a white, crystalline precipitate of magnesium ammonium phosphate will indicate the presence of magnesium salts.

(10) *Sulphates*, which may at times be present as magnesium sulphate (Epsom's salts), may be detected by adding a few drops of hydrochloric acid to a portion of the extract, followed by the addition of a few drops of a solution of barium chloride. The formation of a white precipitate of barium sulphate will indicate the presence of sulphates.

(11) *Resins, fats, and oils* may be tested for by the methods already given in the preceding experiments.

(12) *China clay* (aluminium silicate), *gypsum* (calcium sulphate), and *talc* (magnesium silicate) are insoluble, even in strong acids, and may be found in the ash left from the ignition of a portion of the fabric, after first boiling in water to remove all soluble matters. It will hardly be necessary here to discriminate between these three substances themselves, as such an analysis would be both tedious and complicated.

**Experiment 182. Determination of the Nature of Mordants on Woolen Fabrics.** — To determine the character of the mordant which may be present in a yarn or fabric, a qualitative analysis of the ash must be made. For this purpose, take about 10 grams of the clippings of the sample and ignite, in small portions at a time, thoroughly in a porcelain crucible until all the carbon and volatile matters have been burned away. The different mordants are then tested for in the following manner:

(1) *Aluminium compounds.* — The ash should be white, or grayish. Dissolve a portion in warm hydrochloric acid, and neutralize the solution with a slight excess of ammonia. A white precipitate of aluminium hydrate will indicate the presence of aluminium. This should be confirmed by heating a portion of the ash on charcoal, moistening with a drop of cobalt nitrate solution; a blue color will be obtained if aluminium is present.

(2) *Tin compounds.* — The ash is also white or grayish. Dissolve in boiling hydrochloric acid, and test a portion of the solution with sulphuretted hydrogen gas. The formation of a brownish yellow precipitate will indicate the presence of tin.

(3) *Iron compounds.* — The ash is reddish brown in color. Dissolve in warm hydrochloric acid. To a portion of the solution add a drop of nitric acid and a few drops of a solution of potassium ferrocyanide (yellow prussiate). The formation of a blue precipitate will indicate the presence of iron. This may be further confirmed by taking another portion of the solution, adding a drop of nitric acid, and a few drops of a solution of potassium sulphocyanide. The formation of a red color indicates the presence of iron.



(4) *Chromium compounds.* — The ash is yellowish or brownish green. Add a few crystals of potassium chlorate and fuse. The resulting yellowish mass is dissolved in water; a few drops of acetic acid are added, and also a solution of lead acetate. A yellow precipitate of lead chromate will indicate the presence of chromium.

(5) *Copper compounds.* — The ash may be brownish or black. Dissolve in warm hydrochloric acid, neutralize with an excess of ammonia water, when the formation of a blue color will indicate the presence of copper.

In some cases, several of these metals may occur together in the ash of the fabric. In order to conduct a systematic test for all of them proceed in the following manner:

Boil up the ash with a little concentrated hydrochloric acid, and filter from any insoluble residue. To the diluted filtrate add hydrogen sulphide until no further precipitation occurs. A precipitate will indicate the presence of copper or tin (consisting of black copper sulphide or brown tin sulphide). Filter, wash the precipitate, and treat with warm ammonium sulphide solution. This will dissolve any tin sulphide and leave the copper sulphide as a residue. Filter the latter, if present; dissolve the residue in a small quantity of strong nitric acid; dilute with water and add slight excess of ammonia, when the formation of a blue color will indicate the presence of **copper**. Tin, if present in the last filtrate, may be identified by adding slight excess of hydrochloric acid and boiling till all odor of hydrogen sulphide is gone. The solution would now contain stannous chloride; filter, and pour into a hot solution of mercuric chloride; a white precipitate of mercurous chloride will indicate the presence of **tin**. The filtrate from the precipitated sulphides of copper and tin is boiled until all odor of hydrogen sulphide is removed. Add a few drops of concentrated nitric acid and boil for a few minutes longer. Add a slight excess of ammonia water and boil again; this will cause the precipitation of any iron, chromium, or aluminium as hydrates. Filter, and wash the residue. Then boil up with a solution of potassium hydrate which will dissolve out any alu-

minium hydrate. Filter from the residue of iron and chromium hydrates; acidify the filtrate with hydrochloric acid, and then add ammonia in slight excess. The formation of a colorless, gelatinous precipitate of aluminium hydrate will indicate the presence of **aluminium**. The above residue of iron and chromium hydrates is now fused on a piece of platinum foil with a small quantity of sodium peroxide. This will result in the formation of sodium chromate, and the fused mass will be yellow if chromium is present. Dissolve the fusion in water; filter off any residue of iron oxide, acidify the filtrate with acetic acid, and add a few drops of lead acetate solution. The formation of a yellow precipitate of lead chromate will indicate the presence of **chromium**. The residue of iron oxide, if present, is dissolved in a little hot concentrated hydrochloric acid; dilute with water and add a few drops of potassium ferrocyanide solution. The formation of a blue precipitate indicates the presence of **iron**.

Analyze samples of cloth containing mordants of

- (a) iron and aluminium.
- (b) iron and chromium.
- (c) tin and aluminium.
- (d) copper and iron.
- (e) iron, aluminium, and copper.
- (f) tin, aluminium, and chromium.

**Experiment 183. Determination of the Nature of Mordants on Cotton Fabrics.** — The mordants which are liable to be present in cotton fabrics fall under three classes:

- (a) Those for basic dyes, including tannin, antimony, iron, and copper.
- (b) Those for alizarin dyes, including tannin, aluminium, iron, chromium, and fatty acids.
- (c) Those for acid dyes, including tannin, aluminium, fatty acids, tin, and lead.

The first class is the principal one, whereas the last two classes rarely come into observation, and then only for a few

specific colors. The various mordants may be detected as follows:

(1) *Tannin*. — A sample of the fabric is boiled in a dilute solution of soda ash for 20 minutes; the solution is then poured off and almost neutralized with hydrochloric acid. A few drops of ferric chloride solution are next added, when the formation of a black color (due to tannate of iron) will indicate the presence of tannin.

(2) *Antimony*. — A sample of the fabric is boiled for 15 minutes with concentrated hydrochloric acid. The solution is filtered and diluted with water. A portion of the filtrate is then treated with hydrogen sulphide, when the formation of a yellow (or orange) precipitate (of antimony sulphide) will indicate the presence of antimony. To confirm this, however, the antimony sulphide is filtered off and dissolved in a little hot concentrated hydrochloric acid. Dilute with water, and add to the solution a piece of zinc on platinum foil. If a black stain forms on the platinum, the presence of antimony is confirmed.

(3) *Iron*. — A portion of the hydrochloric acid extract obtained above is tested with a few drops of potassium ferrocyanide solution. The formation of a blue color will indicate the presence of iron.

(4) *Copper*. — Another portion of the same acid solution as above is neutralized with an excess of ammonia water. The formation of a blue color in the liquid will indicate the presence of copper.

In case the fabric is suspected to have been dyed with alizarin colors (Turkey-red, etc.), *tannin* may be tested for in the manner given above. *Aluminium*, *iron*, *chromium*, and *tin* mordants may be looked for according to the methods given in Exp. 181. *Fatty acids* may be tested for by first boiling the sample with dilute hydrochloric acid (to decompose the fatty acid compounds of the metallic mordants) and then extracting with petroleum ether and testing as in Exp. 176.

When it is suspected that the sample is dyed with acid colors (which may usually be told by the color not being at all fast to



soap and water), and it is desired to identify the mordants used, those of *aluminium*, *tannin*, *fatty acid*, and *tin* may be detected as given above.

(5) *Lead*. — The sample is boiled for 20 minutes in concentrated nitric acid. Dilute the solution, and filter. To a portion of the filtrate add a few drops of a solution of potassium chromate, when the formation of a yellow precipitate of lead chromate will indicate the presence of lead.

### QUIZ 28.

771. What is meant by the sizing and dressing materials on a fabric? How may the amount of these be estimated?

772. What is meant by "conditioning"? What is the meaning of "normal" fibre and "bone-dry" fibre?

773. How is the conditioning test usually made? What is understood by the "regain"? What is the customary regain allowed for woolen, cotton, and silk fabrics?

774. If 100 grams of woolen yarn give 84 grams of bone-dry fibre, how much normal fibre does it contain?

775. A shipment of 12,750 pounds of worsted tops is billed on a basis of 18 per cent. regain; what would be the conditioned weight allowed if a test showed the presence of 16 per cent. of moisture?

776. A firm buys 1125 pounds of raw silk at \$4.72 per pound with 11 per cent. regain. The conditioning test gives 12½ per cent. of moisture; what was the amount of the bill?

777. A cotton mill manufactures 125,000 pounds of cotton yarn per month at an average selling price of 38 cents per pound. This yarn when sold averages 4.2 per cent. moisture. At the end of the year the mill shows a loss of \$12,500. The mill then dampens its yarn so that it contains 8 per cent. moisture. What profit should the mill show at the end of the second year?

778. How is the amount of oil and grease in a fabric determined? Describe the operation of extraction in a Soxhlet apparatus.

779. How is the presence of a mineral oil in a fabric detected?

780. What is a resin oil? How is the presence of such detected in fabrics? How is the presence of resin itself shown?

781. How would you estimate the total amount of mineral matter present in a textile fabric?

782. What are the chief mordants liable to occur in woolen goods? What mordants yield a white ash on ignition?

783. Give the method for testing for aluminium compounds on a woolen fabric. For chromium. For tin.

784. Suppose a sample of woolen cloth contained iron and aluminium mordants; how would you show their presence?

785. If iron, aluminium, and copper compounds were present in a woolen fabric, how would you conduct the analysis?

786. Name the principal ingredients which may be present in the sizing of a textile fabric.

787. Which of the common sizing ingredients are soluble in water and which are not?

788. Give methods for the detection of the following substances in the sizing of fabrics:

Starch

Gelatin

Zinc chloride

Dextrin

Sugar

Epsom's salts

China clay

Calcium chloride

789. What are the chief mordants likely to be present in cotton fabrics?

790. How would you show the presence of tannin mordant on a cotton fabric? Of antimony? Of iron? Of fatty acids?

791. How would you show the presence of lead compounds in a cotton fabric?

## APPENDIX

### USEFUL DATA FOR DYERS AND TEXTILE CHEMISTS

1. **Hydrometers.** — The strength of many solutions is most conveniently measured by a determination of the density. The instrument used for this purpose is known as a hydrometer.

*The Twaddle Hydrometer.* — This is an instrument for measuring the density of solutions and liquids. Each Twaddle degree (abbreviated to Tw.) represents 0.005 units of specific gravity, and the starting point for liquids heavier than water is the density of water, which is 1 sp. gr. and is made equal to 0 degrees Tw. Hence 1° Tw. would be 1.005 sp. gr.; 2° Tw. would be 1.010 sp. gr.; 10° Tw. would be 1.050 sp. gr., etc. To convert specific gravity readings into degrees Twaddle, and *vice versa*, the following formulas may be employed:

$$\begin{aligned}\text{Twaddle degrees} &= (\text{specific gravity} - 1) \times 200; \\ &= \frac{\text{specific gravity} - 1}{0.005}\end{aligned}$$

$$\text{Specific gravity} = (\text{Tw.}^\circ \times 0.005) + 1.$$

*The Baumé Hydrometer.* — This is an instrument very similar to that of Twaddle, but its method of graduation is different. The degrees Baumé (abbreviated to Bé.) bear no direct relation to actual specific gravity, but this hydrometer is largely used for technical work both in Europe and America. The graduation for liquids heavier than water is made in the following manner. The zero mark (as with the Twaddle instrument) is obtained by immersion in distilled water; the instrument is then placed in a solution containing 15 parts by weight of common-salt and 85 parts by weight of water, and the point to which the hydrometer sinks is called 15. The interval between this point and the zero is then divided into 15 equal parts, and the graduation continued as far as desirable. The degrees Baumé represent greater



## COMPARISON BETWEEN THE SPECIFIC GRAVITY OF BEAUMÉ AND TWADDLE

Tw.	B.	Sp. gr.	Tw.	B.	Sp. gr.	Tw.	B.	Sp. gr.
0	0	1.000	58	32.4	1.290	116	53.0	1.580
1	0.7	1.005	59	32.8	1.295	117	53.3	1.585
2	1.4	1.010	60	33.3	1.300	118	53.6	1.590
3	2.1	1.015	61	33.7	1.305	119	53.9	1.595
4	2.7	1.020	62	34.2	1.310	120	54.1	1.600
5	3.4	1.025	63	34.6	1.315	121	54.4	1.605
6	4.1	1.030	64	35.0	1.320	122	54.7	1.610
7	4.7	1.035	65	35.4	1.325	123	55.0	1.615
8	5.4	1.040	66	35.8	1.330	124	55.2	1.620
9	6.0	1.045	67	36.2	1.335	125	55.5	1.625
10	6.7	1.050	68	36.6	1.340	126	55.8	1.630
11	7.4	1.055	69	37.0	1.345	127	56.0	1.635
12	8.0	1.060	70	37.4	1.350	128	56.3	1.640
13	8.7	1.065	71	37.8	1.355	129	56.6	1.645
14	9.4	1.070	72	38.2	1.360	130	56.9	1.650
15	10.0	1.075	73	38.6	1.365	131	57.1	1.655
16	10.6	1.080	74	39.0	1.370	132	57.4	1.660
17	11.2	1.085	75	39.4	1.375	133	57.7	1.665
18	11.9	1.090	76	39.8	1.380	134	57.9	1.670
19	12.4	1.095	77	40.1	1.385	135	58.2	1.675
20	13.0	1.100	78	40.5	1.390	136	58.4	1.680
21	13.6	1.105	79	40.8	1.395	137	58.7	1.685
22	14.2	1.110	80	41.2	1.400	138	58.9	1.690
23	14.9	1.115	81	41.6	1.405	139	59.2	1.695
24	15.4	1.120	82	42.0	1.410	140	59.5	1.700
25	16.0	1.125	83	42.3	1.415	141	59.7	1.705
26	16.5	1.130	84	42.7	1.420	142	60.0	1.710
27	17.1	1.135	85	43.1	1.425	143	60.2	1.715
28	17.7	1.140	86	43.4	1.430	144	60.4	1.720
29	18.3	1.145	87	43.8	1.435	145	60.6	1.725
30	18.8	1.150	88	44.1	1.440	146	60.9	1.730
31	19.3	1.155	89	44.4	1.445	147	61.1	1.735
32	19.8	1.160	90	44.8	1.450	148	61.4	1.740
33	20.3	1.165	91	45.1	1.455	149	61.6	1.745
34	20.9	1.170	92	45.4	1.460	150	61.8	1.750
35	21.4	1.175	93	45.8	1.465	151	62.1	1.755
36	22.0	1.180	94	46.1	1.470	152	62.3	1.760
37	22.5	1.185	95	46.4	1.475	153	62.5	1.765
38	23.0	1.190	96	46.7	1.480	154	62.8	1.770
39	23.5	1.195	97	47.1	1.485	155	63.0	1.775
40	24.0	1.200	98	47.4	1.490	156	63.2	1.780
41	24.5	1.205	99	47.8	1.495	157	63.5	1.785
42	25.0	1.210	100	48.1	1.500	158	63.7	1.790
43	25.5	1.215	101	48.4	1.505	159	64.0	1.795
44	26.0	1.220	102	48.7	1.510	160	64.2	1.800
45	26.4	1.225	103	49.0	1.515	161	64.4	1.805
46	26.9	1.230	104	49.4	1.520	162	64.6	1.810
47	27.4	1.235	105	49.7	1.525	163	64.8	1.815
48	27.9	1.240	106	50.0	1.530	164	65.0	1.820
49	28.4	1.245	107	50.3	1.535	165	65.2	1.825
50	28.8	1.250	108	50.6	1.540	166	65.5	1.830
51	29.3	1.255	109	50.9	1.545	167	65.7	1.835
52	29.7	1.260	110	51.2	1.550	168	65.9	1.840
53	30.2	1.265	111	51.5	1.555	169	66.1	1.845
54	30.6	1.270	112	51.8	1.560	170	66.3	1.850
55	31.1	1.275	113	52.1	1.565	171	66.5	1.855
56	31.5	1.280	114	52.4	1.570	172	66.7	1.860
57	32.0	1.285	115	52.7	1.575	173	67.0	1.865

density than corresponding degrees Twaddle. The table on the previous page shows the equivalence between specific gravity, degrees Twaddle, and degrees Beaumé.

A solution of a certain density may be diluted with water (density 1.000) to a solution of another specified density in accordance with the following formula:

Let  $V$  = volume of strong solution,  
 $v$  = volume of water to be added,  
 $D$  = density of strong solution (in specific gravity),  
 $d$  = density of diluted solution (in specific gravity).

Then 
$$v = V \times \frac{D - d}{d - 1}.$$

If the densities are expressed in degrees Twaddle, the formula becomes:

$$v = V \times \frac{T - t}{t},$$

where  $T$  = density of strong solution in degrees Twaddle.

$t$  = density of diluted solution in degrees Twaddle.

EXAMPLE. — How much water would it be necessary to add to 100 cc. of a solution of sulphuric acid of 1.84 sp. gr. to give a solution of 1.12 sp. gr.?

$$v = 100 \times \frac{1.84 - 1.12}{1.12 - 1} = 600 \text{ cc.}$$

How much water must be added to 1 litre of a solution of caustic soda of 90° Tw. to reduce it to 35° Tw.?

$$\begin{aligned} v &= 1 \times \frac{90 - 35}{35} = 1.571 \text{ litres} \\ &= 1571 \text{ cc.} \end{aligned}$$

**2. Equivalents of Common Use in Measuring.** — In all scientific and accurate work the metric system of weights and measures is universally employed. It is presumed that the student is familiar in a general way with the method and values of the metric system, but his attention is called at this point to following equivalents, both of the metric system and the common English system, which will be found useful and practical for reference.

- 1 litre (*l.*) = 1000 cubic centimetres (*cc.*).  
 1 litre of water weighs 1 kilogram (*kilo.*).  
 1 cc. of water weighs 1 gram (*gm.*).  
 1 cubic foot of water weighs 62.5 pounds.  
 1 gram = 1000 milligrams (*mgm.*).  
 1 kilogram = 1000 grams = 2.2 pounds.  
 1 pound (Avoir.) = 453.9 grams.  
 1 gallon (U. S.) = 231 cubic inches.  
 1 gallon water = 8.3 pounds.  
 1 pint water = 1 pound (approximately).  
 1 litre = 1 quart (approximately).

To convert feet to metres multiply by 0.3.

Metres to feet multiply by 3.3.

Cubic feet to gallons multiply by 7.5.

Gallons to cubic feet multiply by 0.13.

Cubic feet to litres multiply by 28.33.

Litres to cubic feet multiply by 0.035.

Inches to centimeters multiply by 2.5.

Centimeters to inches multiply by 0.4.

Ounces to grams multiply by 28.35.

Grams to ounces multiply by 0.04.

Grains to grams multiply by 0.065.

Grams to grains multiply by 15.43.

Yards to metres multiply by 0.9.

Metres to yards multiply by 1.1.

Quarts to litres multiply by 0.95.

Litres to quarts multiply by 1.06.

Gallons to litres multiply by 3.78.

Litres to gallons multiply by 0.26.

An English (Imperial) gallon is larger than the United States gallon; it contains  $277\frac{1}{4}$  cubic inches = 4.54 litres; it contains 10 pounds of water.

#### CONVERSION OF KILOGRAMS INTO POUNDS.

Kilos.	Lbs.	Kilos.	Lbs.	Kilos.	Lbs.	Kilos.	Lbs.
1	$2\frac{1}{8}$	7	$15\frac{1}{2}$	40	88	100	$220\frac{1}{2}$
2	$4\frac{5}{8}$	8	$17\frac{5}{8}$	50	$110\frac{1}{4}$	200	441
3	$6\frac{3}{8}$	9	$19\frac{1}{4}$	60	132	300	$661\frac{1}{2}$
4	$8\frac{7}{8}$	10	$22\frac{1}{4}$	70	154	400	882
5	11	20	$44\frac{1}{4}$	80	176	500	$1102\frac{1}{2}$
6	$13\frac{1}{8}$	30	$66\frac{3}{8}$	90	198	600	1323



## CONVERSION OF POUNDS INTO KILOGRAMS.

Lbs.	Kilos.	Lbs.	Kilos.	Lbs.	Kilos.	Lbs.	Kilos.
1	0.453	11	4.984	21	9.515	40	18.125
2	0.906	12	5.437	22	9.968	50	22.656
3	1.359	13	5.890	23	10.421	60	27.187
4	1.812	14	6.343	24	10.874	70	31.719
5	2.265	15	6.796	25	11.327	80	36.250
6	2.719	16	7.249	26	11.780	90	40.781
7	3.172	17	7.702	27	12.233	100	45.302
8	3.625	18	8.155	28	12.686	200	90.625
9	4.078	19	8.608	29	13.139	300	135.937
10	4.531	20	9.062	30	13.594	400	181.250

## CONVERSION OF OUNCES INTO GRAMS.

Ozs.	Grams.	Ozs.	Grams.	Ozs.	Grams.	Ozs.	Grams.
1	28.35	5	141.75	9	255.15	13	368.54
2	56.70	6	170.10	10	283.50	14	396.89
3	85.05	7	198.45	11	311.84	15	425.24
4	113.40	8	226.80	12	340.19	16	453.59

## CONVERSION OF GRAMS INTO OUNCES AND GRAINS.

Grams.	Ozs.	Grains.	Grams.	Ozs.	Grains.	Grams.	Ozs.	Grains.
1	.....	15.43	23	.....	355	44	1	241
2	.....	30.9	24	.....	370	45	1	257
3	.....	46.3	25	.....	386	46	1	272
4	.....	61.7	26	.....	401	47	1	288
5	.....	77.2	27	.....	417	48	1	303
6	.....	92.6	28	.....	432	49	1	319
7	.....	108	29	1	10	50	1	334
8	.....	123	30	1	25	51	1	350
9	.....	139	31	1	41	52	1	365
10	.....	154	32	1	56	53	1	380
11	.....	170	33	1	72	54	1	395
12	.....	185	34	1	87	55	1	411
13	.....	201	35	1	102	56	1	427
14	.....	216	36	1	118	57	2	5
15	.....	231	37	1	133	58	2	20
16	.....	247	38	1	149	59	2	36
17	.....	262	39	1	164	60	2	51
18	.....	278	40	1	180	70	2	205
19	.....	293	41	1	195	80	2	360
20	.....	309	42	1	210	90	3	76
21	.....	324	43	1	226	100	3	230
22	.....	340						

### 3. Thermometry. Comparison of Centigrade Thermometer with Fahrenheit.

Deg. Cent.	Deg. Fahr.	Deg. Cent.	Deg. Fahr.	Deg. Cent.	Deg. Fahr.	Deg. Cent.	Deg. Fahr.
110	230	80	176.	50	122.	20	68.
109	228.2	79	174.2	49	120.2	19	66.2
108	226.4	78	172.4	48	118.4	18	64.4
107	224.6	77	170.6	47	116.6	17	62.6
106	222.8	76	168.8	46	114.8	16	60.8
105	221.	75	167.	45	113.	15	59.
104	219.2	74	165.2	44	111.2	14	57.2
103	217.4	73	163.4	43	109.4	13	55.4
102	215.6	72	161.6	42	107.6	12	53.6
101	213.8	71	159.8	41	105.8	11	51.8
100	212.	70	158.	40	104.	10	50.
99	210.2	69	156.2	39	102.2	9	48.2
98	208.4	68	154.4	38	100.4	8	46.4
97	206.6	67	152.6	37	98.6	7	44.6
96	204.8	66	150.8	36	96.8	6	42.8
95	203.	65	149.	35	95.	5	41.
94	201.2	64	147.2	34	93.2	4	39.2
93	199.4	63	145.4	33	91.4	3	37.4
92	197.6	62	143.6	32	89.6	2	35.6
91	195.8	61	141.8	31	87.8	1	33.8
90	194.	60	140.	30	86.	0	32.
89	192.2	59	138.2	29	84.2	-1	30.2
88	190.4	58	136.4	28	82.4	2	28.4
87	188.6	57	134.6	27	80.6	3	26.6
86	186.8	56	132.8	26	78.8	4	24.8
85	185.	55	131.	25	77.	5	23.
84	183.2	54	129.2	24	75.2	6	21.2
83	181.4	53	127.4	23	73.4	7	19.4
82	179.6	52	125.6	22	71.6	8	17.6
81	177.8	51	123.8	21	69.8	9	15.8

To convert degrees Centigrade to degrees Fahrenheit:

$$(C^{\circ} \times 9) \div 5 \text{ and add } 32 = F^{\circ}.$$

To convert degrees Fahrenheit to degrees Centigrade:

$$(F^{\circ} - 32) \times 5 \div 9 = C^{\circ}.$$

### 4. Comparison of Relative Strengths of Chemicals.

100 parts by weight of *sal soda* are equivalent to 37 parts of *soda ash*.

100 parts of *soda ash* are equivalent to 270 parts of *sal soda*.

100 parts of *crystallized glauber salt* are equivalent to 44 parts of *calcined glauber salt*.

100 parts of *calcined glauber salt* are equivalent to 227 parts of *crystallized glauber salt*.

100 parts of *alum* are equivalent in dyeing value to 60 parts of *aluminium sulphate*.

100 parts of *aluminium sulphate* are equivalent to 170 parts of *alum*.

100 parts of *sulphuric acid* 168° Tw. correspond to 220 parts *hydrochloric acid* 32° Tw., and to 400 parts *acetic acid* 9° Tw.

100 parts of *hydrochloric acid* 32° Tw. correspond to 45 parts of *sulphuric acid* 168° Tw., and to 175 parts of *acetic acid* 9° Tw.

100 parts of *acetic acid* 9° Tw. correspond to 26 parts of *sulphuric acid* 168° Tw., and to 57 parts of *hydrochloric acid* 32° Tw.

100 parts of *crystallized sodium sulphide* are equivalent to 50 parts of *concentrated sodium sulphide*.

100 parts of *concentrated sodium sulphide* are equivalent to 200 parts of the *crystallized*.

## 5. Tables Showing the Strengths and Densities of Various Solutions.

### SULPHURIC ACID.

At 60° F. (15° C.).

Deg. Tw.	Per cent. Sulphuric Acid.	Deg. Tw.	Per cent. Sulphuric Acid.	Deg. Tw.	Per cent. Sulphuric Acid.	Deg. Tw.	Per cent. Sulphuric Acid.
2	1.57	48	32.28	94	56.90	140	77.17
4	3.03	50	33.43	96	57.83	142	78.04
6	4.49	52	34.57	98	58.74	144	78.92
8	5.96	54	35.71	100	59.70	146	79.80
10	7.37	56	36.87	102	60.65	148	80.68
12	8.77	58	38.03	104	61.59	150	81.56
14	10.19	60	39.19	106	62.53	152	82.44
16	10.90	62	40.35	108	63.43	154	83.32
18	12.99	64	41.50	110	64.26	156	84.50
20	14.35	66	42.66	112	65.08	158	85.70
22	15.71	68	43.74	114	65.90	160	86.90
24	17.01	70	44.82	116	66.71	162	88.30
26	18.31	72	45.88	118	67.59	164	90.05
28	19.61	74	46.94	120	68.51	165	91.00
30	20.91	76	48.50	122	69.43	166	92.10
32	22.19	78	49.06	124	70.32	167	93.43
34	23.47	80	50.11	126	71.16	168	95.60
36	24.76	82	51.15	128	71.99	168.3*	97.70
38	26.04	84	52.15	130	72.82	168.1	98.70
40	27.32	86	53.11	132	73.64	168	99.20
42	28.58	88	54.07	134	74.51	167.7	99.95
44	29.84	90	55.03	136	75.42	.....	.....
46	31.11	92	55.97	138	76.30	.....	.....

\* Sulphuric acid of 97.70 per cent. has the highest density, whilst that of the stronger acid is slightly lower.



## HYDROCHLORIC ACID.

At 60° F.

Deg. Tw.	Per cent. Hydro- chloric Acid.	Deg. Tw.	Per cent. Hydro- chloric Acid.	Deg. Tw.	Per cent. Hydro- chloric Acid.	Deg. Tw.	Per cent. Hydro- chloric Acid.
1	1.15	11	11.18	21	20.97	31	30.55
2	2.14	12	12.19	22	21.92	32	31.52
3	3.12	13	13.19	23	22.86	33	32.49
4	4.13	14	14.17	24	23.82	34	33.46
5	5.15	15	15.16	25	24.78	35	34.42
6	6.15	16	16.15	26	25.75	36	35.39
7	7.15	17	17.13	27	26.70	37	36.31
8	8.16	18	18.11	28	27.66	38	37.23
9	9.16	19	19.06	29	28.61	39	38.16
10	10.17	20	20.01	30	29.57	40	39.11

From this table it will be seen that the degree Twaddle indicates approximately the percentage of hydrochloric acid in the solution.

## ACETIC ACID.

At 60° F.

Per cent. Acetic Acid.	Deg. Tw.	Per cent. Acetic Acid.	Deg. Tw.	Per cent. Acetic Acid.	Deg. Tw.	Per cent. Acetic Acid.	Deg. Tw.
5	1.3	30	8.2	55	13.1	80	15.0
10	2.8	35	9.4	60	13.7	85	14.8
15	4.3	40	10.5	65	14.3	90	14.3
20	5.7	45	11.4	70	14.7	95	13.2
25	7.0	50	12.3	75	14.9	100	11.1

The densities above 11° Tw. correspond to two liquids of different strengths. To determine if the solution corresponds to the stronger or the weaker acid, a small quantity of water is added, and the density is again measured. If the density increases on addition of water the acid is the stronger; whereas if it diminishes the acid is the weaker.

## CAUSTIC SODA.

At 60° F.

Per cent. Caustic Soda.	Deg. Tw.	Per cent. Caustic Soda.	Deg. Tw.	Per cent. Caustic Soda.	Deg. Tw.	Per cent. Caustic Soda.	Deg. Tw.
1	2.4	16	36.2	31	68.6	46	99.8
2	4.6	17	38.4	32	70.2	47	101.6
3	7.0	18	40.4	33	72.6	48	103.8
4	9.2	19	42.6	34	74.8	49	105.8
5	11.8	20	45.0	35	76.8	50	108.0
6	14.0	21	47.2	36	79.0	51	110.0
7	16.2	22	49.4	37	81.0	52	112.0
8	18.4	23	51.6	38	83.0	53	114.0
9	20.6	24	53.8	39	85.2	54	116.0
10	23.0	25	55.8	40	87.4	55	118.2
11	25.2	26	58.0	41	89.4	56	120.2
12	27.4	27	60.0	42	91.5	57	122.2
13	29.6	28	62.0	43	93.6	58	124.4
14	31.8	29	64.2	44	95.6	59	126.6
15	34.0	30	66.4	45	97.6	60	128.6

## SODA ASH.

At 60° F.

Deg. Tw.	Per cent. Sodium Carbonate.	Deg. Tw.	Per cent. Sodium Carbonate.	Deg. Tw.	Per cent. Sodium Carbonate.	Deg. Tw.	Per cent. Sodium Carbonate.
1	0.47	9	4.28	17	8.04	25	11.76
2	0.95	10	4.76	18	8.51	26	12.23
3	1.42	11	5.23	19	8.97	27	12.70
4	1.90	12	5.71	20	9.43	28	13.16
5	2.38	13	6.17	21	9.90	29	13.63
6	2.85	14	6.64	22	10.37	30	14.09
7	3.33	15	7.10	23	10.83	.....	.....
8	3.80	16	7.57	24	11.30	.....	.....

## GLAUBERSALT.

At 66° F.

Per cent. Glaubersalt.	Sp. gr.	Per cent. Glaubersalt.	Sp. gr.	Per cent. Glaubersalt.	Sp. gr.	Per cent. Glaubersalt.	Sp. gr.
1	1.0040	9	1.0358	17	1.0683	25	1.1015
2	1.0079	10	1.0398	18	1.0725	26	1.1057
3	1.0118	11	1.0439	19	1.0766	27	1.1100
4	1.0158	12	1.0479	20	1.0807	28	1.1142
5	1.0198	13	1.0520	21	1.0849	29	1.1184
6	1.0238	14	1.0560	22	1.0890	30	1.1226
7	1.0278	15	1.0601	23	1.0931	.....	.....
8	1.0318	16	1.0642	24	1.0973	.....	.....

The percentage of desiccated (or calcined) glaubersalt,  $\text{Na}_2\text{SO}_4$ , may be obtained by multiplying the above percentages of crystallized glaubersalt by the factor 0.441.

## COMMON SALT (SODIUM CHLORIDE).

At 60° F.

Per cent. Sodium Chloride.	Sp. gr.	Per cent. Sodium Chloride.	Sp. gr.	Per cent. Sodium Chloride.	Sp. gr.	Per cent. Sodium Chloride.	Sp. gr.
1	1.00725	8	1.05851	15	1.11146	22	1.16755
2	1.01450	9	1.06593	16	1.11938	23	1.17580
3	1.02174	10	1.07335	17	1.12730	24	1.18404
4	1.02899	11	1.08097	18	1.13523	25	1.19228
5	1.03624	12	1.08859	19	1.14315	26	1.20098
6	1.04366	13	1.09622	20	1.15107	26.4	1.20433
7	1.05108	14	1.10384	21	1.15931	.....	.....

## TANNIC ACID.

At 60° F.

Per cent. Tannic Acid.	Sp. gr.	Per cent. Tannic Acid.	Sp. gr.	Per cent. Tannic Acid.	Sp. gr.	Per cent. Tannic Acid.	Sp. gr.
1.0	1.0040	2.1	1.0084	3.2	1.0128	4.3	1.0172
1.1	1.0044	2.2	1.0088	3.3	1.0132	4.4	1.0176
1.2	1.0048	2.3	1.0092	3.4	1.0136	4.5	1.0180
1.3	1.0052	2.4	1.0096	3.5	1.0140	4.6	1.0184
1.4	1.0056	2.5	1.0100	3.6	1.0144	4.7	1.0188
1.5	1.0060	2.6	1.0104	3.7	1.0148	4.8	1.0192
1.6	1.0064	2.7	1.0108	3.8	1.0152	4.9	1.0196
1.7	1.0068	2.8	1.0112	3.9	1.0156	5.0	1.0200
1.8	1.0072	2.9	1.0116	4.0	1.0160	.....	.....
1.9	1.0076	3.0	1.0120	4.1	1.0164	.....	.....
2.0	1.0080	3.1	1.0124	4.2	1.0168	.....	.....



## BLEACHING POWDER (CHLORIDE OF LIME).

At 60° F.

Density.		Available chlorine.		
Sp. gr.	Tw. deg.	Per litre.	Per gallon.	
		Grains.	Ozs.	Grains.
I. 1155	23. I	71. 79	II	213
I. 1150	23	71. 50	II	193
I. 1105	22. I	68. 66	10	431
I. 1100	22	68. 00	10	385
I. 1060	21. 2	65. 33	10	198
I. 1050	21	64. 50	10	140
I. 1000	20	61. 17	9	346
I. 0950	19	58. 33	9	146
I. 0900	18	55. 18	8	363
I. 0850	17	52. 27	8	159
I. 0800	16	48. 96	7	365
I. 0750	15	45. 70	7	137
I. 0700	14	42. 31	6	337
I. 0650	13	38. 71	6	85
I. 0600	12	35. 81	5	320
I. 0550	11	32. 68	5	101
I. 0500	10	29. 41	4	309
I. 0450	9	26. 62	4	113
I. 0400	8	23. 75	3	351
I. 0350	7	20. 44	3	119
I. 0300	6	17. 36	2	340
I. 0250	5	14. 47	2	137
I. 0200	4	11. 44	1	362
I. 0150	3	8. 48	1	157
I. 0100	2	5. 58	.....	391
I. 0050	1	2. 71	.....	190
I. 0025	$\frac{1}{2}$	1. 40	.....	98

PROPORTIONS OF CHLORINE IN WEAK SOLUTIONS  
OF BLEACHING POWDER.

Deg. Tw.	Effective chlorine, grams per litre.
3	8. 48
$1\frac{1}{2}$	2. 05
1	2. 71
$\frac{3}{4}$	4. 15

## 6. — Tables for Calculations in Dyeing.

PERCENTAGE OF DYESTUFF CORRESPONDING TO GRAMS PER 100 KILOS,  
AND POUNDS PER 100 POUNDS OF GOODS.

Per cent.	Per 100 kilo.	Per 100 lbs.		Per cent.	Per 100 kilo.	Per 100 lbs.		Per cent.	Per 100 kilo.	Per 100 lbs.	
	gms.	lb. oz.	grns.		gms.	lb. oz.	grns.		gms.	lb. oz.	grns.
0.001	1		7	0.29	290	4	280	0.65	650	10	175
0.002	2		14	0.30	300	4	350	0.66	660	10	245
0.003	3		21	0.31	310	4	420	0.67	670	10	315
0.004	4		28	0.32	320	5	53	0.68	680	10	385
0.005	5		35	0.33	330	5	123	0.69	690	11	18
0.006	6		42	0.34	340	5	193	0.70	700	11	88
0.007	7		49	0.35	350	5	263	0.71	710	11	158
0.008	8		56	0.36	360	5	333	0.72	720	11	228
0.009	9		63	0.37	370	5	403	0.73	730	11	298
0.01	10		70	0.38	380	6	35	0.74	740	11	368
0.02	20		140	0.39	390	6	105	0.75	750	12	...
0.03	30		210	0.40	400	6	175	0.76	760	12	70
0.04	40		280	0.41	410	6	245	0.77	770	12	140
0.05	50		350	0.42	420	6	315	0.78	780	12	210
0.06	60		420	0.43	430	6	385	0.79	790	12	280
0.07	70	1	53	0.44	440	7	18	0.80	800	12	350
0.08	80	1	123	0.45	450	7	88	0.81	810	12	420
0.09	90	1	193	0.46	460	7	158	0.82	820	13	53
0.10	100	1	263	0.47	470	7	228	0.83	830	13	123
0.11	110	1	333	0.48	480	7	298	0.84	840	13	193
0.12	120	1	403	0.49	490	7	368	0.85	850	13	263
0.13	130	2	35	0.50	500	8	...	0.86	860	13	333
0.14	140	2	106	0.51	510	8	70	0.87	870	13	403
0.15	150	2	176	0.52	520	8	140	0.88	880	14	35
0.16	160	2	246	0.53	530	8	210	0.89	890	14	105
0.17	170	2	316	0.54	540	8	280	0.90	900	14	175
0.18	180	2	386	0.55	550	8	350	0.91	910	14	245
0.19	190	3	18	0.56	560	8	420	0.92	920	14	315
0.20	200	3	88	0.57	570	9	53	0.93	930	14	385
0.21	210	3	158	0.58	580	9	123	0.94	940	15	18
0.22	220	3	229	0.59	590	9	193	0.95	950	15	88
0.23	230	3	299	0.60	600	9	263	0.96	960	15	158
0.24	240	3	369	0.61	610	9	333	0.97	970	15	228
0.25	250	4	...	0.62	620	9	403	0.98	980	15	298
0.26	260	4	70	0.63	630	10	35	0.99	990	15	368
0.27	270	4	140	0.64	640	10	105	1	1 Kilo	1	
0.28	280	4	210								

1 lb. = 16 oz. = 7000 grains = 454 grams.

1 oz. = 437½ grains = 28.349 grams.

1 gram = 15.43 grains.

COMPARISON OF DYE-TESTS WITH TEST-SKEINS OF 5 GRAMS (77 GRAINS)  
AND PRACTICAL DYEING OF 100 POUNDS MATERIAL.

The standard solutions for the dye-tests contain 1 gram of dyestuff dissolved in 1 litre of water.

For 5 grams samples.	Equiva- lent per- centage.	Equivalent per 100 lbs.			For 5 grams samples.	Equiva- lent per- centage.	Equivalent per 100 lbs.		
ccm.	per cent.	lb.	oz.	grns.	ccm.	per cent.	lb.	oz.	grns.
1	0.02			140	47	0.94		15	18
2	0.04		$\frac{1}{2}$	62	48	0.96		15	158
3	0.06		$\frac{1}{2}$	202	49	0.98		15 $\frac{1}{2}$	179
4	0.08	1		123	50	1.00	I		
5	0.10	1 $\frac{1}{2}$		44	51	1.02	I		140
6	0.12	1 $\frac{1}{2}$		184	52	1.04	I	$\frac{1}{2}$	62
7	0.14	2		105	53	1.06	I	$\frac{1}{2}$	202
8	0.16	2 $\frac{1}{2}$		27	54	1.08	I	1	123
9	0.18	2 $\frac{1}{2}$		167	55	1.10	I	1 $\frac{1}{2}$	44
10	0.20	3		88	56	1.12	I	1 $\frac{1}{2}$	184
11	0.22	3 $\frac{1}{2}$		9	57	1.14	I	2	105
12	0.24	3 $\frac{1}{2}$		199	58	1.16	I	2 $\frac{1}{2}$	27
13	0.26	4		70	59	1.18	I	2 $\frac{1}{2}$	167
14	0.28	4		210	60	1.20	I	3	88
15	0.30	4 $\frac{1}{2}$		132	61	1.22	I	3 $\frac{1}{2}$	9
16	0.32	5		53	62	1.24	I	3 $\frac{1}{2}$	149
17	0.34	5		193	63	1.26	I	4	70
18	0.36	5 $\frac{1}{2}$		114	64	1.28	I	4	210
19	0.38	6		35	65	1.30	I	4 $\frac{1}{2}$	132
20	0.40	6		175	66	1.32	I	5	53
21	0.42	6 $\frac{1}{2}$		97	67	1.34	I	5	193
22	0.44	7		18	68	1.36	I	5 $\frac{1}{2}$	114
23	0.46	7		158	69	1.38	I	6	35
24	0.48	7 $\frac{1}{2}$		79	70	1.40	I	6	175
25	0.50	8		00	71	1.42	I	6 $\frac{1}{2}$	97
26	0.52	8		140	72	1.44	I	7	13
27	0.54	8 $\frac{1}{2}$		62	73	1.46	I	7	158
28	0.56	8 $\frac{1}{2}$		202	74	1.48	I	7 $\frac{1}{2}$	79
29	0.58	9		123	75	1.50	I	8	
30	0.60	9 $\frac{1}{2}$		44	76	1.52	I	8	140
31	0.62	9 $\frac{1}{2}$		184	77	1.54	I	8 $\frac{1}{2}$	62
32	0.64	10		105	78	1.56	I	8 $\frac{1}{2}$	202
33	0.66	10 $\frac{1}{2}$		27	79	1.58	I	9	123
34	0.68	10 $\frac{1}{2}$		167	80	1.60	I	9 $\frac{1}{2}$	44
35	0.70	11		88	81	1.62	I	9 $\frac{1}{2}$	184
36	0.72	11 $\frac{1}{2}$		9	82	1.64	I	10	105
37	0.74	11 $\frac{1}{2}$		149	83	1.66	I	10 $\frac{1}{2}$	27
38	0.76	12		70	84	1.68	I	10 $\frac{1}{2}$	167
39	0.78	12		210	85	1.70	I	11	88
40	0.80	12 $\frac{1}{2}$		132	86	1.72	I	11 $\frac{1}{2}$	9
41	0.82	13		53	87	1.74	I	11 $\frac{1}{2}$	149
42	0.84	13		193	88	1.76	I	12	70
43	0.86	13 $\frac{1}{2}$		114	89	1.78	I	12	210
44	0.88	14		35	90	1.80	I	12 $\frac{1}{2}$	132
45	0.90	14		175	91	1.82	I	13	53
46	0.92	14 $\frac{1}{2}$		97	92	1.84	I	13	193



## COMPARISON OF DYE-TESTS. — Continued.

For 5 grams samples.	Equiva- lent per- centage.	Equivalent per 100 lbs.			For 5 grams samples.	Equiva- lent per- centage.	Equivalent per 100 lbs.		
ccm.	per cent.	lb.	oz.	grns.	ccm.	per cent.	lb.	oz.	grns.
93	1.86	1	13½	114	147	2.94	2	15	18
94	1.88	1	14	35	148	2.96	2	15	158
95	1.90	1	14	175	149	2.98	2	15½	179
96	1.92	1	14½	97	150	3.00	3	0	
97	1.94	1	15	18	151	3.02	3	0	140
98	1.96	1	15	158	152	3.04	3	½	62
99	1.98	1	15½	79	153	3.06	3	½	202
100	2.00	2			154	3.08	3	1	123
101	2.02	2		140	155	3.10	3	1½	44
102	2.04	2	½	62	156	3.12	3	1½	184
103	2.06	2	½	202	157	3.14	3	2	105
104	2.08	2	1	123	158	3.16	3	2½	27
105	2.10	2	1½	44	159	3.18	3	2½	167
106	2.12	2	1½	184	160	3.20	3	3	88
107	2.14	2	2	105	161	3.22	3	3½	9
108	2.16	2	2½	27	162	3.24	3	3½	149
109	2.18	2	2½	167	163	3.26	3	4	70
110	2.20	2	3	88	164	3.28	3	4	210
111	2.22	2	3½	9	165	3.30	3	4½	132
112	2.24	2	3½	149	166	3.32	3	5	53
113	2.26	2	4	70	167	3.34	3	5	193
114	2.28	2	4	210	168	3.36	3	5½	114
115	2.30	2	4½	132	169	3.38	3	6	35
116	2.32	2	5	53	170	3.40	3	6	175
117	2.34	2	5	193	171	3.42	3	6½	97
118	2.36	2	5½	114	172	3.44	3	7	18
119	2.38	2	6	35	173	3.46	3	7	158
120	2.40	2	6	175	174	3.48	3	7½	79
121	2.42	2	6½	97	175	3.50	3	8	
122	2.44	2	7	185	176	3.52	3	8	140
123	2.46	2	7	158	177	3.54	3	8½	62
124	2.48	2	7½	79	178	3.56	3	8½	202
125	2.50	2	8		179	3.58	3	9	123
126	2.52	2	8	140	180	3.60	3	9½	44
127	2.54	2	8½	62	181	3.62	3	9½	184
128	2.56	2	8½	202	182	3.64	3	10	105
129	2.58	2	9	123	183	3.66	3	10½	27
130	2.60	2	9½	44	184	3.68	3	10½	167
131	2.62	2	9½	184	185	3.70	3	11	88
132	2.64	2	10	105	186	3.72	3	11½	9
133	2.66	2	10½	27	187	3.74	3	11½	149
134	2.68	2	10½	167	188	3.76	3	12	70
135	2.70	2	11	88	189	3.78	3	12	210
136	2.72	2	11½	9	190	3.80	3	12½	132
137	2.74	2	11½	149	191	3.82	3	13	53
138	2.76	2	12	70	192	3.84	3	13	193
139	2.78	2	12	210	193	3.86	3	13½	114
140	2.80	2	12½	132	194	3.88	3	14	35
141	2.82	2	13	53	195	3.90	3	14	175
142	2.84	2	13	193	196	3.92	3	14½	97
143	2.86	2	13½	114	197	3.94	3	15	18
144	2.88	2	14	35	198	3.96	3	15	158
145	2.90	2	14	175	199	3.98	3	15½	79
146	2.92	2	14½	97	200	4.00	4		

## REDUCTION OF GRAMS TO OUNCES PER 100 LBS. GOODS.

Grams.	Ounces for solu- tion.	Grams.	Ounces for solu- tion.	Grams.	Ounces for solu- tion.
1	.35	11	3.88	21	7.41
2	.71	12	4.24	22	7.76
3	1.06	13	4.58	23	8.12
4	1.41	14	4.93	24	8.47
5	1.77	15	5.29	25	8.82
6	2.12	16	5.64	26	9.17
7	2.47	17	5.99	27	9.53
8	2.83	18	6.34	28	9.88
9	3.18	19	6.60	29	10.22
10	3.53	20	7.06	30	10.59

To use any number of grams per 100 pounds of goods, dissolve the corresponding number of ounces in above table in 10 gallons of water, and use 1 gallon of this solution per 100 pounds goods.

FOR EXAMPLE. To use 42 grams of dyestuff per 100 pounds goods:

$$30 \text{ grams} = 10.59 \text{ ounces}$$

$$10 \text{ grams} = 3.53 \text{ ounces}$$

$$\frac{2 \text{ grams}}{30} = \frac{.71}{10} \text{ ounces}$$

$$42 \text{ grams} = 14.83 \text{ ounces}$$

Therefore, dissolve 14.83 ounces of dyestuff in 10 gallons of water and use 1 gallon of the solution.

*To Determine the Capacity of a Rectangular Vat.* — Multiply the length by the breadth by the depth in feet to obtain the cubic contents; then multiply this number by  $7\frac{1}{2}$  to obtain the capacity in gallons (United States).

Or, multiply the length by the breadth by the depth in inches, and divide the result by 231 to obtain the capacity in gallons.

*To Determine the Capacity of a Round Vat.* — Multiply the diameter in feet by itself; multiply the result by the factor 0.7854, and then by the depth in feet; finally multiply this result by  $7\frac{1}{2}$  to obtain the capacity in gallons.

Or, multiply the diameter in inches by itself, then multiply by 0.7854, and then by the depth in inches. Divide the result by 231 to obtain the capacity in gallons.

REDUCTION OF GRAMS PER KILOGRAM OF GOODS TO OUNCES  
PER 100 POUNDS OF GOODS.

Grams per kilo.	Ounces per 100 lbs.	Grams per kilo.	Ounces per 100 lbs.	Grams per kilo.	Ounces per 100 lbs.
1	1.6	18	28.8	35	56.0
2	3.2	19	30.4	36	57.6
3	4.8	20	32.0	37	59.2
4	6.4	21	33.6	38	60.8
5	8.0	22	35.2	39	62.4
6	9.6	23	36.8	40	64.0
7	11.2	24	38.4	41	65.6
8	12.8	25	40.0	42	67.2
9	14.4	26	41.6	43	68.8
10	16.0	27	43.2	44	70.4
11	17.6	28	44.8	45	72.0
12	19.2	29	46.4	46	73.6
13	20.8	30	48.0	47	75.2
14	22.4	31	49.6	48	76.8
15	24.0	32	51.2	49	78.4
16	25.6	33	52.8	50	80.0
17	27.2	34	54.4	.....	.....

To convert grams per kilo. into ounces per 100 lbs., multiply by the factor 1.6.

REDUCTION OF FRACTIONAL PERCENTAGES TO OUNCES PER 100 POUNDS  
OF GOODS.

Per cent.	Ounces per 100 lbs. goods.	Per cent.	Ounces per 100 lbs. goods.	Per cent.	Ounces per 100 lbs. goods.	Per cent.	Ounces per 100 lbs. goods.
$\frac{1}{2}$	8.00	$\frac{1}{8}$	2.00	$\frac{5}{11}$	7.28	$\frac{9}{13}$	11.11
$\frac{1}{3}$	5.33	$\frac{3}{8}$	6.00	$\frac{6}{11}$	8.73	$\frac{10}{13}$	12.31
$\frac{2}{3}$	10.66	$\frac{5}{8}$	10.00	$\frac{7}{11}$	10.19	$\frac{11}{13}$	13.54
$\frac{1}{4}$	4.00	$\frac{7}{8}$	14.00	$\frac{8}{11}$	11.64	$\frac{12}{13}$	14.77
$\frac{3}{4}$	12.00	$\frac{1}{9}$	1.77	$\frac{9}{11}$	13.09	$\frac{1}{14}$	1.14
$\frac{1}{5}$	3.20	$\frac{2}{9}$	3.55	$\frac{10}{11}$	14.55	$\frac{2}{14}$	3.44
$\frac{2}{5}$	6.40	$\frac{4}{9}$	7.11	$\frac{1}{12}$	1.34	$\frac{3}{14}$	5.72
$\frac{3}{5}$	9.60	$\frac{5}{9}$	8.88	$\frac{1}{12}$	6.66	$\frac{4}{14}$	10.29
$\frac{4}{5}$	12.80	$\frac{7}{9}$	12.44	$\frac{1}{12}$	9.33	$\frac{5}{14}$	10.57
$\frac{1}{6}$	2.66	$\frac{8}{9}$	14.22	$\frac{1}{12}$	14.66	$\frac{6}{14}$	14.86
$\frac{5}{6}$	13.30	$\frac{1}{10}$	1.60	$\frac{1}{13}$	1.23	$\frac{7}{15}$	1.07
$\frac{1}{7}$	2.29	$\frac{2}{10}$	4.80	$\frac{2}{13}$	2.46	$\frac{8}{15}$	2.13
$\frac{2}{7}$	4.58	$\frac{3}{10}$	11.20	$\frac{3}{13}$	3.69	$\frac{9}{15}$	4.27
$\frac{3}{7}$	6.86	$\frac{4}{10}$	14.40	$\frac{4}{13}$	4.92	$\frac{10}{15}$	6.40
$\frac{4}{7}$	9.15	$\frac{5}{10}$	1.45	$\frac{5}{13}$	6.16	$\frac{11}{15}$	7.47
$\frac{5}{7}$	11.43	$\frac{6}{10}$	2.91	$\frac{6}{13}$	7.39	$\frac{12}{15}$	8.53
$\frac{6}{7}$	13.80	$\frac{7}{10}$	4.37	$\frac{7}{13}$	8.61	$\frac{13}{15}$	11.73
		$\frac{8}{10}$	5.82	$\frac{8}{13}$	9.85	$\frac{14}{15}$	13.87
						$\frac{15}{15}$	14.96



## REDUCTION OF DECIMAL PERCENTAGES TO OUNCES PER 100 POUNDS OF GOODS.

Per cent.	Ounces per 100 lbs. goods.	Per cent.	Ounces per 100 lbs. goods.	Per cent.	Ounces per 100 lbs. goods.	Per cent.	Ounces per 100 lbs. goods.
.10	1.60	.35	5.60	.60	9.60	.85	13.60
.15	2.40	.40	6.40	.65	10.40	.90	14.40
.20	3.20	.45	7.20	.70	11.20	.95	15.20
.25	4.00	.50	8.00	.75	12.00	1.00	16.00
.30	4.80	.55	8.80	.80	12.80	.....	.....

## REDUCTION OF DECIMAL PARTS OF POUNDS TO OUNCES.

Lbs.	Oz.	Lbs.	Oz.	Lbs.	Oz.	Lbs.	Oz.
.01	.16	.14	2.24	.27	4.32	.40	6.40
.02	.32	.15	2.40	.28	4.48	.41	6.56
.03	.48	.16	2.56	.29	4.64	.42	6.72
.04	.64	.17	2.72	.30	4.80	.43	6.88
.05	.80	.18	2.88	.31	4.96	.44	7.04
.06	.96	.19	3.04	.32	5.12	.45	7.20
.07	1.12	.20	3.20	.33	5.28	.46	7.36
.08	1.28	.21	3.36	.34	5.44	.47	7.52
.09	1.44	.22	3.52	.35	5.60	.48	7.68
.10	1.60	.23	3.68	.36	5.76	.49	7.84
.11	1.76	.24	3.84	.37	5.92	.50	8.00
.12	1.92	.25	4.00	.38	6.08	.....	.....
.13	2.08	.26	4.16	.39	6.24	.....	.....

## REDUCTION OF DECIMAL PERCENTAGES OF GALLONS TO QUARTS AND PINTS.

Gals.	Qts.	Pts.	Gals.	Qts.	Pts.	Gals.	Qts.	Pts.
.05	.....	$\frac{2}{5}$	.40	1	$1\frac{1}{5}$	.75	3	.....
.10	.....	$\frac{4}{5}$	.45	1	$1\frac{3}{5}$	.80	3	$\frac{2}{5}$
.15	.....	$1\frac{1}{5}$	.50	2	.....	.85	3	$\frac{4}{5}$
.20	.....	$1\frac{2}{5}$	.55	2	$2\frac{2}{5}$	.90	3	$1\frac{1}{5}$
.25	1	.....	.60	2	$2\frac{4}{5}$	.95	3	$1\frac{3}{5}$
.30	1	$2\frac{2}{5}$	.65	2	$1\frac{1}{5}$	1.00	4	.....
.35	1	$\frac{4}{5}$	.70	2	$1\frac{3}{5}$	.....	.....	.....

REDUCTION OF LITRES PER KILOGRAM OF GOODS TO GALLONS PER  
100 POUNDS OF GOODS.

Litres per kilo.	Gallons per 100 pounds.	Litres per kilo.	Gallons per 100 pounds.	Litres per kilo.	Gallons per 100 pounds.	Litres per kilo.	Gallons per 100 pounds.
1	11.99	14	167.88	27	323.78	40	479.68
2	23.98	15	179.88	28	335.77	41	491.67
3	35.97	16	191.87	29	347.76	42	503.66
4	47.96	17	203.86	30	359.76	43	515.65
5	59.96	18	215.85	31	371.75	44	527.64
6	71.95	19	227.84	32	383.74	45	539.64
7	83.94	20	239.84	33	395.73	46	551.63
8	95.93	21	251.83	34	407.72	47	563.62
9	107.92	22	263.82	35	419.72	48	575.61
10	119.92	23	275.81	36	431.71	49	587.60
11	131.91	24	287.80	37	443.70	50	599.60
12	143.90	25	299.80	38	455.69	.....	.....
13	155.89	26	311.79	39	467.68	.....	.....

REDUCTION OF GRAMS PER LITRE TO OUNCES PER GALLON.

Grams per litre.	Ounces per gallon.	Grams per litre.	Ounces per gallon.	Grams per litre.	Ounces per gallon.	Grams per litre.	Ounces per gallon.
1	.13	9	1.20	17	2.26	25	3.33
2	.26	10	1.33	18	2.40	26	3.46
3	.40	11	1.46	19	2.53	27	3.60
4	.53	12	1.60	20	2.66	28	3.73
5	.66	13	1.73	21	2.80	29	3.86
6	.80	14	1.86	22	2.93	30	4.00
7	.93	15	2.00	23	3.06	.....	.....
8	1.06	16	2.13	24	3.20	.....	.....

To convert grams per litre into ounces per gallon, multiply by the factor 0.133.

PERCENTAGE TABLES.

Per cent.	For 10 pounds.	For 50 pounds.	For 100 pounds.
10.0	1 lb.	5 lbs.	10 lbs.
9.0	14 oz. 175 grains	4½ lbs.	9 lbs.
8.0	12 oz. 350 grains	4 lbs.	8 lbs.
7.0	11 oz. 87 grains	3½ lbs.	7 lbs.
6.0	9 oz. 263 grains	3 lbs.	6 lbs.
5.0	8 oz.	2½ lbs.	5 lbs.
4.0	6 oz. 175 grains	2 lbs.	4 lbs.
3.0	4 oz. 350 grains	1½ lbs.	3 lbs.
2.0	3 oz. 88 grains	1 lb.	2 lbs.
1.0	1 oz. 263 grains	8 oz.	1 lb.
0.99	1 oz. 256 grains	7 oz. 403 grains	15 oz. 368 grains
0.98	1 oz. 249 grains	7 oz. 368 grains	15 oz. 298 grains
0.97	1 oz. 242 grains	7 oz. 333 grains	15 oz. 228 grains
0.96	1 oz. 235 grains	7 oz. 298 grains	15 oz. 158 grains
0.95	1 oz. 228 grains	7 oz. 263 grains	15 oz. 88 grains
0.94	1 oz. 221 grains	7 oz. 228 grains	15 oz. 18 grains
0.93	1 oz. 214 grains	7 oz. 193 grains	14 oz. 385 grains
0.92	1 oz. 207 grains	7 oz. 158 grains	14 oz. 315 grains
0.91	1 oz. 200 grains	7 oz. 123 grains	14 oz. 245 grains
0.90	1 oz. 193 grains	7 oz. 88 grains	14 oz. 175 grains
0.89	1 oz. 186 grains	7 oz. 53 grains	14 oz. 105 grains
0.88	1 oz. 179 grains	7 oz. 18 grains	14 oz. 35 grains
0.87	1 oz. 172 grains	6 oz. 420 grains	13 oz. 403 grains
0.86	1 oz. 165 grains	6 oz. 385 grains	13 oz. 333 grains
0.85	1 oz. 158 grains	6 oz. 350 grains	13 oz. 263 grains
0.84	1 oz. 151 grains	6 oz. 315 grains	13 oz. 193 grains
0.83	1 oz. 144 grains	6 oz. 280 grains	13 oz. 123 grains
0.82	1 oz. 137 grains	6 oz. 245 grains	13 oz. 53 grains
0.81	1 oz. 130 grains	6 oz. 210 grains	12 oz. 420 grains
0.80	1 oz. 123 grains	6 oz. 175 grains	12 oz. 350 grains
0.79	1 oz. 116 grains	6 oz. 140 grains	12 oz. 280 grains
0.78	1 oz. 109 grains	6 oz. 105 grains	12 oz. 210 grains
0.77	1 oz. 102 grains	6 oz. 70 grains	12 oz. 140 grains
0.76	1 oz. 95 grains	6 oz. 35 grains	12 oz. 70 grains
0.75	1 oz. 88 grains	6 oz.	12 oz.
0.74	1 oz. 81 grains	5 oz. 403 grains	11 oz. 368 grains
0.73	1 oz. 74 grains	5 oz. 368 grains	11 oz. 298 grains
0.72	1 oz. 67 grains	5 oz. 333 grains	11 oz. 228 grains
0.71	1 oz. 60 grains	5 oz. 298 grains	11 oz. 158 grains
0.70	1 oz. 53 grains	5 oz. 263 grains	11 oz. 88 grains



PERCENTAGE TABLES.

Per cent.	For 10 pounds.	For 50 pounds.	For 100 pounds.
0.69	1 oz. 46 grains	5 oz. 228 grains	11 oz. 18 grains
0.68	1 oz. 39 grains	5 oz. 193 grains	10 oz. 385 grains
0.67	1 oz. 32 grains	5 oz. 158 grains	10 oz. 315 grains
0.66	1 oz. 25 grains	5 oz. 123 grains	10 oz. 245 grains
0.65	1 oz. 18 grains	5 oz. 88 grains	10 oz. 175 grains
0.64	1 oz. 11 grains	5 oz. 53 grains	10 oz. 105 grains
0.63	1 oz. 4 grains	5 oz. 18 grains	10 oz. 35 grains
0.62	434 grains	4 oz. 420 grains	9 oz. 403 grains
0.61	427 grains	4 oz. 385 grains	9 oz. 333 grains
0.60	420 grains	4 oz. 350 grains	9 oz. 263 grains
0.59	413 grains	4 oz. 315 grains	9 oz. 193 grains
0.58	406 grains	4 oz. 280 grains	9 oz. 123 grains
0.57	399 grains	4 oz. 245 grains	9 oz. 53 grains
0.56	392 grains	4 oz. 210 grains	8 oz. 420 grains
0.55	385 grains	4 oz. 175 grains	8 oz. 350 grains
0.54	378 grains	4 oz. 140 grains	8 oz. 280 grains
0.53	371 grains	4 oz. 105 grains	8 oz. 210 grains
0.52	364 grains	4 oz. 70 grains	8 oz. 140 grains
0.51	357 grains	4 oz. 35 grains	8 oz. 70 grains
0.50	350 grains	4 oz.	8 oz.
0.49	343 grains	3 oz. 403 grains	7 oz. 368 grains
0.48	336 grains	3 oz. 368 grains	7 oz. 298 grains
0.47	329 grains	3 oz. 333 grains	7 oz. 228 grains
0.46	322 grains	3 oz. 298 grains	7 oz. 158 grains
0.45	315 grains	3 oz. 263 grains	7 oz. 88 grains
0.44	308 grains	3 oz. 228 grains	7 oz. 18 grains
0.43	301 grains	3 oz. 193 grains	6 oz. 385 grains
0.42	294 grains	3 oz. 158 grains	6 oz. 315 grains
0.41	287 grains	3 oz. 123 grains	6 oz. 245 grains
0.40	280 grains	3 oz. 88 grains	6 oz. 175 grains
0.39	273 grains	3 oz. 53 grains	6 oz. 105 grains
0.38	266 grains	3 oz. 18 grains	6 oz. 35 grains
0.37	259 grains	2 oz. 420 grains	5 oz. 403 grains
0.36	252 grains	2 oz. 385 grains	5 oz. 333 grains
0.35	245 grains	2 oz. 350 grains	5 oz. 263 grains
0.34	238 grains	2 oz. 315 grains	5 oz. 193 grains
0.33	231 grains	2 oz. 280 grains	5 oz. 123 grains
0.32	224 grains	2 oz. 245 grains	5 oz. 53 grains
0.31	217 grains	2 oz. 210 grains	4 oz. 420 grains
0.30	210 grains	2 oz. 175 grains	4 oz. 350 grains

PERCENTAGE TABLES.

Per cent.	For 10 pounds.	For 50 pounds.	For 100 pounds.
0.29	203 grains	2 oz. 140 grains	4 oz. 280 grains
0.28	196 grains	2 oz. 105 grains	4 oz. 210 grains
0.27	189 grains	2 oz. 70 grains	4 oz. 140 grains
0.26	182 grains	2 oz. 35 grains	4 oz. 70 grains
0.25	175 grains	2 oz.	4 oz.
0.24	168 grains	1 oz. 403 grains	3 oz. 368 grains
0.23	161 grains	1 oz. 368 grains	3 oz. 298 grains
0.22	154 grains	1 oz. 333 grains	3 oz. 228 grains
0.21	147 grains	1 oz. 298 grains	3 oz. 158 grains
0.20	140 grains	1 oz. 263 grains	3 oz. 88 grains
0.19	133 grains	1 oz. 228 grains	3 oz. 18 grains
0.18	126 grains	1 oz. 193 grains	2 oz. 385 grains
0.17	119 grains	1 oz. 158 grains	2 oz. 315 grains
0.16	112 grains	1 oz. 123 grains	2 oz. 245 grains
0.15	105 grains	1 oz. 88 grains	2 oz. 175 grains
0.14	98 grains	1 oz. 53 grains	2 oz. 105 grains
0.13	91 grains	1 oz. 18 grains	2 oz. 35 grains
0.12	84 grains	420 grains	1 oz. 403 grains
0.11	77 grains	385 grains	1 oz. 333 grains
0.10	70 grains	350 grains	1 oz. 263 grains
0.09	63 grains	315 grains	1 oz. 193 grains
0.08	56 grains	280 grains	1 oz. 123 grains
0.07	49 grains	245 grains	1 oz. 53 grains
0.06	42 grains	210 grains	420 grains
0.05	35 grains	175 grains	350 grains
0.04	28 grains	140 grains	280 grains
0.03	21 grains	105 grains	210 grains
0.02	14 grains	70 grains	140 grains
0.01	7 grains	35 grains	70 grains

The following example will illustrate the use of this table: How much dyestuff would be required for 2.23 per cent. on 70 pounds of material?

{ For 50 lbs. 2% equals 1 lb.  
 { For 20 lbs. 2% equals 6 oz. 176 grains  
 { For 50 lbs. 0.23% equals 1 oz. 368 grains  
 { For 20 lbs. 0.23% equals 322 grains

For 70 lbs. 2.23% equals 1 lb. 8 oz. 429 grains

TABLE SHOWING THE AMOUNTS OF SODIUM NITRITE, ACID, AND DEVELOPER REQUIRED FOR DIAZOTIZING.

Dyestuff. Per cent.	Sodium nitrite. Per cent.	Sulphuric acid 168° Tw. Per cent.	Or hydrochloric acid in place of sulphuric. Per cent.	Developer. Per cent.
$\frac{1}{2}$	1	2	3	0.5
1	$1\frac{1}{2}$	$2\frac{1}{2}$	$3\frac{1}{2}$	0.6
$1\frac{1}{2}$	$1\frac{1}{2}$	3	$4\frac{1}{2}$	0.7
2	$1\frac{1}{2}$	$3\frac{1}{2}$	5	0.8
$2\frac{1}{2}$	2	4	6	0.9
3	2	4	6	1.
$3\frac{1}{2}$	$2\frac{1}{2}$	5	$7\frac{1}{2}$	1.
4	$2\frac{1}{2}$	5	$7\frac{1}{2}$	1.
$4\frac{1}{2}$	$2\frac{1}{2}$	5	$7\frac{1}{2}$	1.
5	$2\frac{1}{2}$	5	$7\frac{1}{2}$	1.

These figures are not supposed to be in exact chemical proportion, but for practical reasons a sufficient excess of developer is prescribed. Good results are to be obtained from these quantities only when the proportion of dyed material to water is 1 : 15.

TABLE OF ATOMIC WEIGHTS OF PRINCIPAL ELEMENTS.

O = 16.

Element.	Sym- bol.	At. wt.	Element.	Sym- bol.	At. wt.
Aluminium.....	Al	27.1	Magnesium.....	Mg	24.36
Antimony.....	Sb	120.	Manganese.....	Mn	55.
Arsenic....	As	75.	Mercury.....	Hg	203.
Barium.....	Ba	137.4	Molybdenum.....	Mo	96.
Bismuth.....	Bi	208.5	Nickel.....	Ni	58.7
Boron.....	B	11.	Nitrogen.....	N	14.04
Bromine.....	Br	79.96	Oxygen.....	O	16.
Cadmium.....	Cd	112.4	Phosphorus.....	P	31.
Calcium.....	Ca	40.	Platinum.....	Pt	194.8
Carbon.....	C	12.	Potassium.....	K	39.15
Cerium.....	Ce	140.	Silicon.....	Si	28.4
Chlorine.....	Cl	35.5	Silver.....	Ag	107.93
Chromium.....	Cr	52.1	Sodium.....	Na	23.05
Cobalt.....	Co	59.	Strontium.....	Sr	87.6
Copper.....	Cu	63.6	Sulphur.....	S	32.06
Fluorine.....	Fl	19.	Tin.....	Sn	118.5
Gold.....	Au	197.2	Titanium.....	Ti	48.
Hydrogen.....	H	1.01	Tungsten.....	W	184.
Iodine.....	I	126.85	Uranium.....	U	239.5
Iron.....	Fe	56.	Vanadium.....	V	51.2
Lead.....	Pb	206.9	Zinc.....	Zn	65.4



TABLE OF FORMULA AND MOLECULAR WEIGHTS OF PRINCIPAL  
CHEMICALS USED IN DYEING.

Name.	Formula.	Mol. wt.
Acetate of alumina.....	$Al_2(C_2H_3O_2)_2$	408
Acetate of ammonia.....	$NH_4C_2H_3O_2$	77
Acetate of chrome (basic).....	$Cr_2(C_2H_3O_2)_4 \cdot (OH)_2$	374
Acetate of chrome (normal).....	$Cr_2(C_2H_3O_2)_6$	458
Acetate of lime.....	$Ca(C_2H_3O_2)_2$	158
Acetate of nickel.....	$Ni(C_2H_3O_2)_2$	177
Acetate of soda.....	$NaC_2H_3O_2 \cdot 3 H_2O$	136
Acetate of tin.....	$Sn(C_2H_3O_2)_2$	237
Acetic acid.....	$CH_3 \cdot COOH$	60
Aectine.....	$C_8H_5(C_2H_3O_2)_3$	218
Acid sodium sulphate.....	$NaHSO_4$	120
Acid sodium sulphite.....	$NaHSO_3$	104
Alcohol.....	$C_2H_5OH$	46
Alpha-naphthylamine.....	$C_{10}H_7NH_2$	143
Alum (potash).....	$Al_2(SO_4)_3K_2SO_4 \cdot 24 H_2O$	949
Aluminium chloride.....	$Al_2Cl_6$	267
Aluminium sulpho-acetate.....	$Al_2SO_4(C_2H_3O_2)_4$	386
Ammonia.....	$NH_3$	17
Ammonium chloride.....	$NH_4Cl$	54
Ammonium tin chloride.....	$SnCl_4 \cdot 2 NH_4Cl$	367
Ammonium vanadate.....	$(NH_4)_3VO_4$	169
Aniline.....	$C_6H_5NH_2$	93
Aniline salt.....	$C_6H_5NH_2 \cdot HCl$	130
Antimony fluoride.....	$SbF_3$	177
Antimony oxide.....	$Sb_2O_3$	288
Antimony salt.....	$SbF_3(NH_4)_2SO_4$	309
Antimony sodium fluoride.....	$SbF_3NaF$	219
Barium chloride.....	$BaCl_2 \cdot 2 H_2O$	244
Benzene.....	$C_6H_6$	78
Beta-naphthol.....	$C_{10}H_7 \cdot OH$	144
Bichromate of soda.....	$Na_2Cr_2O_7 \cdot 2 H_2O$	298
Bichromate of potash.....	$K_2Cr_2O_7$	295
Bisulphite of chrome.....	$Cr_2(HSO_3)_6$	591
Borax.....	$Na_2B_4O_7 \cdot 10 H_2O$	382
Calcium chloride.....	$CaCl_2$	111
Caustic lime.....	$CaO$	56
Caustic soda.....	$NaOH$	40
Caustic potash.....	$KOH$	56
Cerium chloride.....	$CeCl_3$	246
Chalk.....	$CaCO_3$	100
Chlorate of alumina.....	$Al_2(ClO_3)_6$	555
Chloride of chrome (basic).....	$Cr_2Cl_2(OH)_4$	243
Chlorate of potash.....	$KClO_3$	123
Chlorate of sodium.....	$NaClO_3$	107
Chromate of chrome.....	$Cr_2(CrO_4)_3$	453
Chromate of lead.....	$PbCrO_4$	323
Chrome alum.....	$Cr_2(SO_4)_3K_2SO_4 \cdot 24 H_2O$	999
Chrome oxide.....	$Cr_2O_3$	152
Chromium nitro-acetate.....	$Cr_2(NO_3)_3(C_2H_3O_2)_3$	467

TABLE OF FORMULA AND MOLECULAR WEIGHTS OF PRINCIPAL CHEMICALS USED IN DYEING.—Continued.

Name.	Formula.	Mol. wt.
Common-salt.....	NaCl	59
Cupric chloride.....	$\text{CuCl}_2 \cdot 2 \text{H}_2\text{O}$	171
Double chloride of tin.....	$\text{SnCl}_4 \cdot 3 \text{H}_2\text{O}$	314
Ferric acetate.....	$\text{Fe}_2(\text{C}_2\text{H}_3\text{O}_2)_6$	466
Ferric chloride.....	$\text{Fe}_2\text{Cl}_6$	325
Ferrous acetate.....	$\text{Fe}(\text{C}_2\text{H}_3\text{O}_2)_2$	174
Ferrous chloride.....	$\text{FeCl}_2$	127
Ferrous sulphate.....	$\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$	278
Fluoride of chrome.....	$\text{Cr}_2\text{F}_6 \cdot 8 \text{H}_2\text{O}$	362
Glaubersalt.....	$\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$	322
Glycerin.....	$\text{C}_3\text{H}_5(\text{OH})_3$	92
Hydrate of alumina.....	$\text{Al}_2(\text{OH})_6$	541
Hydrochloric acid.....	HCl	36
Hydrofluoric acid.....	HF	20
Hyposulphite of soda.....	$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$	248
Lactic acid.....	$\text{C}_3\text{H}_5\text{O}_3$	90
Magnesium chloride.....	$\text{MgCl}_2 \cdot 6 \text{H}_2\text{O}$	203
Manganese chloride.....	$\text{MnCl}_2 \cdot 4 \text{H}_2\text{O}$	198
Nitrate of chrome.....	$\text{Cr}_2(\text{NO}_3)_6$	476
Nitrate of lead.....	$\text{Pb}(\text{NO}_3)_2$	331
Nitric acid.....	$\text{HNO}_3$	63
Oxalate of ammonia.....	$(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$	142
Oxalate of antimony.....	$\text{Sb}(\text{C}_2\text{O}_4\text{K})_3 \cdot 6 \text{H}_2\text{O}$	610
Oxalic acid.....	$\text{C}_2\text{O}_4\text{H}_2 \cdot 2 \text{H}_2\text{O}$	126
Oxide of lead.....	$\text{PbO}$	223
Paranitraniline.....	$\text{C}_6\text{H}_4(\text{NO}_2)\text{NH}_2$	138
Permanganate of potash.....	$\text{KMnO}_4$	158
Peroxide of hydrogen.....	$\text{H}_2\text{O}_2$	34
Phenol.....	$\text{C}_6\text{H}_5\text{OH}$	94
Phosphate of soda.....	$\text{Na}_2\text{HPO}_4 \cdot 12 \text{H}_2\text{O}$	354
Potash.....	$\text{K}_2\text{CO}_3 \cdot 2 \text{H}_2\text{O}$	174
Potassium oxalate.....	$\text{KHC}_2\text{O}_4$	128
Red prussiate.....	$\text{K}_4\text{Fe}_2(\text{CN})_{12}$	659
Resorcline.....	$\text{C}_6\text{H}_4(\text{OH})_2$	110
Silicate of soda.....	$\text{Na}_2\text{Si}_2\text{O}_6$	304
Sugar of lead.....	$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3 \text{H}_2\text{O}$	379
Sulphate of alumina.....	$\text{Al}_2(\text{SO}_4)_3 \cdot 18 \text{H}_2\text{O}$	667
Sulphate of cadmium.....	$\text{CdSO}_4 \cdot 2 \text{H}_2\text{O}$	244
Sulphate of copper.....	$\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$	250
Sulphate of lead.....	$\text{PbSO}_4$	302
Sulphate of magnesium.....	$\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$	247
Sulphate of nickel.....	$\text{NiSO}_4 \cdot 7 \text{H}_2\text{O}$	281
Sulphate of zinc.....	$\text{ZnSO}_4 \cdot 7 \text{H}_2\text{O}$	288
Sulphocyanide of ammonia.....	$\text{NH}_4\text{SCN}$	76
Sulphocyanide of copper.....	$\text{Cu}(\text{SCN})_2$	180
Sulphocyanide of iron.....	$\text{Fe}(\text{SCN})_2$	172
Sulphocyanide of potash.....	$\text{KSCN}$	97
Sulphuric acid (Oil of vitriol).....	$\text{H}_2\text{SO}_4$	98

TABLE OF FORMULA AND MOLECULAR WEIGHTS OF PRINCIPAL  
CHEMICALS USED IN DYEING. — Continued.

Name.	Formula.	Mol. wt.
Sulphurous acid.....	$\text{SO}_2$	64
Soda calcined (soda ash) .....	$\text{Na}_2\text{CO}_3$	106
Soda crystallized .....	$\text{Na}_2\text{CO}_3 \cdot 10 \text{ H}_2\text{O}$	286
Sodium aluminate.....	$\text{Na}_6\text{Al}_2\text{O}_6$	289
Sodium bisulphite.....	$\text{NaHSO}_3$	104
Sodium hydrosulphite crystallized.....	$\text{Na}_2\text{S}_2\text{O}_4 \cdot 2 \text{ H}_2\text{O}$	194
Sodium nitrite.....	$\text{NaNO}_2$	69
Sodium peroxide.....	$\text{Na}_2\text{O}_2$	78
Sodium sulphide crystallized.....	$\text{Na}_2\text{S} \cdot 9 \text{ H}_2\text{O}$	240
Stannate of soda.....	$\text{Na}_2\text{SnO}_3$	213
Stannic hydrate.....	$\text{SnO}(\text{OH})_2$	169
Stannous hydrate.....	$\text{Sn}(\text{OH})_2$	153
Tannin.....	$\text{C}_{14}\text{H}_{10}\text{O}_9$	322
Tartar.....	$\text{KH}(\text{C}_4\text{H}_4\text{O}_6)$	188
Tartar emetic.....	$\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot \frac{1}{2} \text{ H}_2\text{O}$	332
Tartar substitute.....	$\text{NaHSO}_4$	120
Tartaric acid.....	$\text{C}_4\text{H}_6\text{O}_6$	150
Thiosulphate of soda .....	$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{ H}_2\text{O}$	248
Tin chloride.....	$\text{SnCl}_4$	260
Tin salt.....	$\text{SnCl}_2 \cdot 2 \text{ H}_2\text{O}$	225
Tungstate of soda.....	$\text{Na}_2\text{WO}_4 \cdot 2 \text{ H}_2\text{O}$	330
Water.....	$\text{H}_2\text{O}$	18
Yellow prussiate .....	$\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3 \text{ H}_2\text{O}$	423
Zinc chloride.....	$\text{ZnCl}_2$	136





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